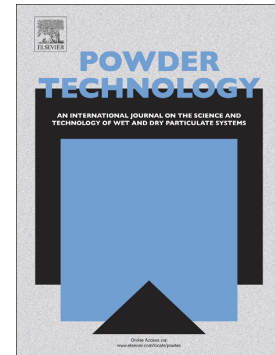


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Diffuse reflectance spectroscopy for the qualification of powder blends for additive manufacturing

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Abstract:

Additive manufacturing (AM) offers significant opportunities for alloy development through direct manufacturing from powder blends. The quality of the AM process is heavily influenced by the properties of the powders used, particularly their homogeneity and flowability. This study evaluates two Al5xxx series powder blends with ZrB₂, CaB₆, and Ta additives for morphology, flowability, rheology, and diffuse reflectance. While flowability and rheological metrics, such as those derived from rotating drum and FT4 tests, showed variability across different blends, their correlations were limited by base powder morphology differences. In contrast, diffuse reflectance spectroscopy (DRS) reliably detected additive type and weight ratios with a normalized difference of only 2.4% between Al5052-additive and Al5254-additive blends, despite their distinct morphologies. This highlights DRS as a non-destructive tool for quality control in powder blend homogeneity. Furthermore, its potential for in situ monitoring in PBF machines could provide real-time blend uniformity data, enabling layer defect detection.

Keywords:

additive manufacturing; metal powder blends; flowability; rheology; reflectance; powder bed fusion

1. Introduction

Additive manufacturing (AM) is a transformative technology that provides flexibility in the production of complex parts [1]. It also enables the in situ formation of new materials through direct fabrication from novel formulations, such as powder blends [2], resin blends [3], or filament composites [4]. Among the various AM techniques, powder bed fusion (PBF), as defined in ISO/ASTM 52900, offers an efficient method for producing modified alloys. This is achieved through the direct processing of powder blends consisting of a base alloy and an alloying addition in the form of an additive powder, facilitating in situ alloying or producing metal matrix composites (MMC). The selection and use of additives in AM are guided by material requirements, particle size and morphology considerations, and compositional targets, supported by theoretical calculations and thermodynamic simulations [5], [6]. This approach has been effectively used in PBF for various purposes, such as refining the grain structure in stainless steel [7], creating functionally graded titanium composites [8], and addressing issues with crack-prone aluminum alloys [9]. Numerous studies in recent years have focused on the in situ functionalization of crack-prone aluminum alloy powders, aiming at grain refinement. The blends introduce functional additives that melt along with the base aluminum powder during the PBF process. These additives form the Al_3X phase during solidification, which enhances the mechanical properties and mitigates cracking tendencies. Commonly used additives include zirconium [10], [11], scandium [12], titanium [13], and their compounds [14], [15].

A variety of techniques for introducing additives into the PBF process have been explored, ranging from high-speed blending [16] and ball milling [17] to 2D rotation blending [18]. The choice of method often depends on the particle size of the additives, which can vary from the base powder scale [18], [19] to the nanometer level [16], and the required amount of additive, which can range from less than 1 wt.% [2] to nearly half of the base powder volume [19]. The additive is selected based on the desired effect: (i) for metal matrix composites (MMCs), larger particles and higher powder volumes are preferred; (ii) in situ alloying requires smaller, homogeneously distributed particles designed to melt during processing; (iii) nanoscale particles are often used to create a near-coating effect, acting as nucleates for grain refinement. Inconsistent powder properties, such as poor particle dispersion or non-uniform additive distribution, can lead to heterogeneous microstructures [19], conglomerate

formation [20], and unpredictable mechanical properties in the final product [21]. The blending process can also lead to loss of the additive concentration [22]. Therefore, proper control of the blending processing parameters is critical to producing homogeneous powder blends that directly influence the microstructure and overall performance of the resulting alloy.

Differences in powder characteristics, such as morphology, particle size distribution (PSD), and bulk density, significantly influence key powder parameters like flowability and rheology. Variations in these properties can lead to inconsistent layer formation during PBF processing and, consequently, defects in the final product [23]. For instance, variations in PSD — a key parameter in PBF — affect both packing density and flowability. Small particles maintain homogeneity until high cohesion sets in, while larger particles achieve optimal homogeneity at moderate cohesion levels [24]. Even minor discrepancies between batches can lead to variations in mechanical properties and microstructures of the final product [25]. These effects are amplified when introducing powder blends into the process. Differences in additive concentration [26], particle size [27], or blending process parameters [28] and blending methods [29], across powder batches, can further reduce flowability, ultimately compromising the overall mechanical performance of the fabricated material through increased porosity [25] or inclusion particle agglomerations [23], which lead to reduced ductility by introducing localized stress concentrators.

Several techniques are commonly used to evaluate the chemical and morphological properties of powders. Methods such as energy dispersive spectroscopy (EDS) [30], X-ray fluorescence (XRF) [31], X-ray diffraction (XRD) [32], combustion elemental analysis (CEA) [33], or inductively coupled plasma (ICP) [34], are frequently employed to determine the chemical composition of metal powders. While these techniques can identify elemental distribution and phase composition, they all have limitations. Many are element specific (e.g., CEA) [33] or struggle with light elements (e.g., EDS, XRF) [35], which limits their ability to provide a comprehensive understanding of multi-component powder blends. Scanning electron microscopy (SEM) is another widely used tool for assessing powder morphology and particle distribution. SEM provides high-resolution images of powder surfaces, making it effective for qualitative analysis [36]. SEM enables quantitative analysis via image segmentation based on contrast criteria [37]. This requires sufficient material density differences for clear contrast

with a backscattered electron detector and a large dataset to minimize sampling error. Moreover, both chemical composition analysis and SEM require specialized equipment and can be costly and time-consuming, which presents challenges in fast-paced manufacturing environments.

In addition to standard material research methods, several powder-oriented characterization techniques are commonly applied to AM metal powders. These include standardized methods such as PSD analysis, Hall funnel measurements, and bulk density (BD) assessments [36], as well as flowability evaluations using cohesion metrics via rotating drum (RD) analyzers [38] or FT4 powder rheometers [39]. PBF-specific in situ [40] and ex situ [41], [42] instruments have also been developed to simulate layer formation in the PBF process, assessing metrics such as flowability and layer density. While these methods effectively measure global powder morphology (e.g., PSD) and flow behavior (e.g., Hall flowmeter, BD, RD, FT4, PBF-specific devices), their ability to assess blend quality in powder mixtures for AM remains underexplored. Recent critical reviews [43], [44], along with experimental studies [45], have examined the capabilities of flowability and spreadability measurement tools, focusing primarily on global powder properties such as particle size, morphology, and flowability, as well as their correlation with layer quality in AM. These works also highlight that these methods are sensitive to different powder characteristics, such as particle size distribution, morphology, and surface texture, which can complicate the correlation of results obtained from various measurement techniques. However, these studies do not evaluate the ability of current tools to assess powder blend quality, underscoring the need for further research in this area.

The optical properties of metal powders have also been employed for the characterization of PBF feedstocks. Although the light absorption of metal powders does not directly translate into melt reflectance in laser-based PBF [46], this type of measurement has proven useful for powder characterization. For example, dual-beam spectroscopy has been used to detect small differences between aged or used powders [47], showing that laser absorption correlates with layer density and thus with part density [25]. Similarly, our previous studies demonstrated that diffuse reflectance spectroscopy (DRS) — an inexpensive method for acquiring the optical properties of materials — can effectively detect powder blends' sensitivity to laser processing parameters [2], identify subtle changes between virgin, used, and spatter samples [34], [48]

and, when properly calibrated, provide quantitative information on powder oxidation and particle size distribution [33]. Therefore, we propose that DRS can also be successfully applied to the assessment of powder blends for additive manufacturing.

The primary goal of this study is to determine whether diffuse reflectance spectroscopy can effectively detect changes in powder blends for additive manufacturing, particularly in identifying additive type, weight proportions, and their influence on powder layer homogeneity. By focusing on Al5xxx series aluminum alloy powders blended with ZrB_2 , CaB_6 , and Ta additives at weight ratios commonly used to functionalize aluminum powders in AM, we explore DRS's potential for assessing compositional homogeneity and powder layer cohesion. By comparing DRS with established techniques such as SEM, bulk density measurement, cohesion analysis, and FT4 rheometry, we demonstrate its effectiveness as a comprehensive tool for layer control in AM processes compared to typically used methods.

2. Materials and methods

2.1. Powders and powder blends

Table 1 provides basic information on the Al5xxx powder blends used in the study. The powder blends are prepared from two types of gas atomized, aluminum powders (Al5052, Al5254) and three selected additive powders (ZrB_2 , CaB_6 or Ta), at selected weight addition ratios (from 45:1 to 150:1). Additive proportions correspond to their expected crack-suppressing effect on Al5xxx alloys processed by laser PBF; however, the thermodynamic formulation rules are beyond the scope of the current study. Al5052 is an aluminum alloy alloyed primarily with 2.2–2.8% magnesium and 0.15–0.35% chromium. Al5254 contains a slightly higher magnesium content of 3.1–3.9% along with 0.15–0.35% chromium. Al5052 is supplied by KAMB Import-Export, Poland, and Al5254 is supplied by Circle Metal Powder Co., Ltd., Taiwan. Both powders have a nominal particle size distribution of 20–63 μm . ZrB_2 , CaB_6 and Ta are supplied by Onyxmet, Poland. The blending process uses a vibratory sieve shaker and ISO 3310:2000 certified analytical sieves (Atest Sp. z o.o., Poland). Aluminum and additive powders are first sieved separately through a 90 μm sieve. The powders are then mixed with additives in a CapsulCN V-1 V-shaped mixer (CapsulCN International Co. Ltd., China) at 30 rpm, changing direction every 5 revolutions, until homogeneity is achieved in 20 minutes. The blend is then

sieved through a 45 μm sieve to remove possible agglomerates. Excess powder is discarded. Each powder blend is based on 100 g of base aluminum powder.

Table 1. Al5xxx powder blends used in the study.

Powder blend	Designation	Addition	Addition nominal d_{10} – d_{90} (μm)	Addition density (g/cm^3)	Addition ratio
Al5052	1	N/A	20–63 (base)	2.68	N/A
Al5052 + ZrB_2	2	ZrB_2	1–10	6.08	50:1
Al5052 + CaB_6	3	CaB_6	10–20	2.45	105:1
Al5052 + CaB_6	4	CaB_6	10–20	2.45	150:1
Al5052 + Ta	5	Ta	5–25	16.65	45:1
Al5052 + Ta	6	Ta	5–25	16.65	60:1
Al5254	A	N/A	20–63 (base)	2.68	N/A
Al5254 + ZrB_2	B	ZrB_2	1–10	6.08	50:1
Al5254 + CaB_6	C	CaB_6	10–20	2.45	105:1
Al5254 + CaB_6	D	CaB_6	10–20	2.45	150:1
Al5254 + Ta	E	Ta	5–25	16.65	45:1
Al5254 + Ta	F	Ta	5–25	16.65	60:1

All powder blending and measurement operations were conducted in a controlled laboratory environment at an ambient temperature of 21 °C and 50% relative humidity. Before and between measurements, the powders were stored in sealed containers with silica gel packets to minimize residual moisture and prevent extensive oxidation.

2.2. Morphology

A Zeiss EVO MA25 scanning electron microscope (SEM) equipped with an EDS detector is used for microscopic observations. Powder samples are collected directly after blending from the bulk of the powder and are mounted on standard 12.5 mm diameter SEM stubs using carbon adhesive tabs. Each powder is sampled three times, at different location within powder volume. PSD is measured using a HELOS BR R4 + RODOS laser diffraction system equipped with VIBRI dispersion unit (Sympatec GmbH, Germany) and analyzed using PAQXOS 3.1 software. Mean diameter (d_{50}) and volumetric mean diameter (VMD) are calculated to characterize the central tendency and volume-weighted mean size of the particles, respectively.

2.3. Flowability

Bulk density is measured using an EV2 volumeter (Electrolab, India) and an XS2002S laboratory balance (Mettler-Toledo, USA). A Hall funnel, specifically a Flow Meter SLM powder funnel (SLM Solutions, Germany) with a non-standard 3.81 mm (0.15 inch) measuring opening and a standard cone angle of $\alpha = 30^\circ$, is used to measure static flowability. For each test, 50 g of powder is used, and a manual stopwatch is employed to measure the flow time. Rotating drum flowability is tested using a GranuDrum material flow analyzer (Granutools, Belgium) in hysteresis mode, where speed increases and then decreases. The analyzer captures 30 images, sampling every 1 second, at rotational speeds of 1, 10, 20, 30, 40, 50, and 60 rpm, starting from 1 rpm. Each powder is measured three times, with a fresh batch used for each measurement. More details about the setup are available in the reference [48]. The flowability of powder samples is characterized using standard rotating drum parameters (cohesive index (CI) and dynamic angle of repose (AOR)). In addition, linear regression was performed, and the fit parameters were determined: the intercept (β_0), the slope (β_1), and the coefficient of determination (R^2) for CI and AOR data.

2.4. Rheology

The rheological properties are evaluated using an FT4 Powder Rheometer (Freeman Technology Ltd, UK). Each powder sample undergoes standardized dynamic flow energy test (stability mode) according to ASTM D7981, including shear, compression, and flow measurements. A 23.5 mm blade and a 25 mm diameter $\times$ 25 ml split vessel are used, with the blade positioned at 55 mm. Prior to measurement, the powder is placed in the vessel and conditioned according to a standardized procedure. After conditioning, the vessel is split to remove excess powder, and the remaining powder is weighed, with the weight measured between 35 and 40 g for all tested powders. The first seven test cycles assess sample stability (stability test sequence) at a constant blade tip speed of 100 mm/s, followed by four cycles evaluating variable flow rates (variable flow rate (VFR) test sequence) at blade tip speeds of 100, 70, 40, and 10 mm/s. Each powder was measured three times, with a fresh batch used for each measurement. The FT4 rheometer is configured to assess basic flowability energy (BFE), defined as the total energy required for the blade to move through the powder under confined flow conditions, using equation 1:

$$BFE = \int_0^h F_N dh \text{ (mJ)}(1),$$

where F_N is the measured normal force and h is the height of the vessel. The stability index (SI) quantifies the consistency of the powder's flowability across repeated test cycles, using equation 2:

$$SI = \frac{BFE_{cycle\ 7}}{BFE_{cycle\ 1}}(2),$$

where the cycle refers to the subsequent iteration of the test. The flow rate index (FRI) measures how the flow energy changes with varying blade tip speeds, using equation 3:

$$FRI = \frac{BFE_{10\ mm/s}}{BFE_{100\ mm/s}}(3),$$

where the index corresponds to the blade tip speed. The specific energy (SE) evaluates the energy required for the blade to ascend through the powder column under unconfined conditions, using equation 4:

$$SE = \frac{BFE_{Unconfined}}{Mass\ of\ Powder} \left(\frac{mJ}{g} \right)(4),$$

The conditioned bulk density (CBD) reflects the powder's bulk density after conditioning, using equation 5:

$$SI = \frac{Split\ Mass}{Split\ Volume} \left(\frac{g}{ml} \right)(5).$$

2.5. Reflectance

Reflectance measurements were performed using diffuse reflectance spectroscopy with an integrating sphere setup employing a d/8° geometry with the specular component included as shown in Figure 1. The setup consisted of an Exemplar Plus BTC655N-ST (1) spectrometer (B&W Tek, USA) and a BIP 2.0 (2) integrating sphere (B&W Tek, USA) with a 20 W tungsten halogen light source. Diffuse illumination was provided, and the detector was positioned at an 8° angle to the sample surface. A BIP 2.0-P PTFE plug ensured that both diffuse and specular reflections were measured. Prior to sample measurements, a dark array acquisition was

performed with the light source turned off. A high-reflectance standard CFWS-99-01c (Avian Technologies LCC, USA) was used as the reference array. Powder samples were prepared in a 20 mm dia. x 10 mm container and levelled (3). The integrating sphere was placed on top of the container. Data were collected in the 900–1100 nm range with a 100 ms integration time and 0.5 nm increments. Each sample measurement was repeated three times, with a fresh batch used for each measurement. Reflectance (%) was calculated using equation 6:

$$\text{Reflectance} = \frac{\text{Raw Array} - \text{Dark Array}}{\text{Reference Array} - \text{Dark Array}} \cdot 100 (\%)(6).$$

To validate the setup, two certified reflectance standards — STONE-AL and FOTOLON — were tested (Instytut Fotonowy Sp. z o.o., Poland). Furthermore, the measurement error for the average reflectance was below 0.01 p.p., as determined by the confidence interval at $\alpha=0.95$ over five consecutive measurements of reflectance standards. The DRS setup used is described in detail in reference [33].

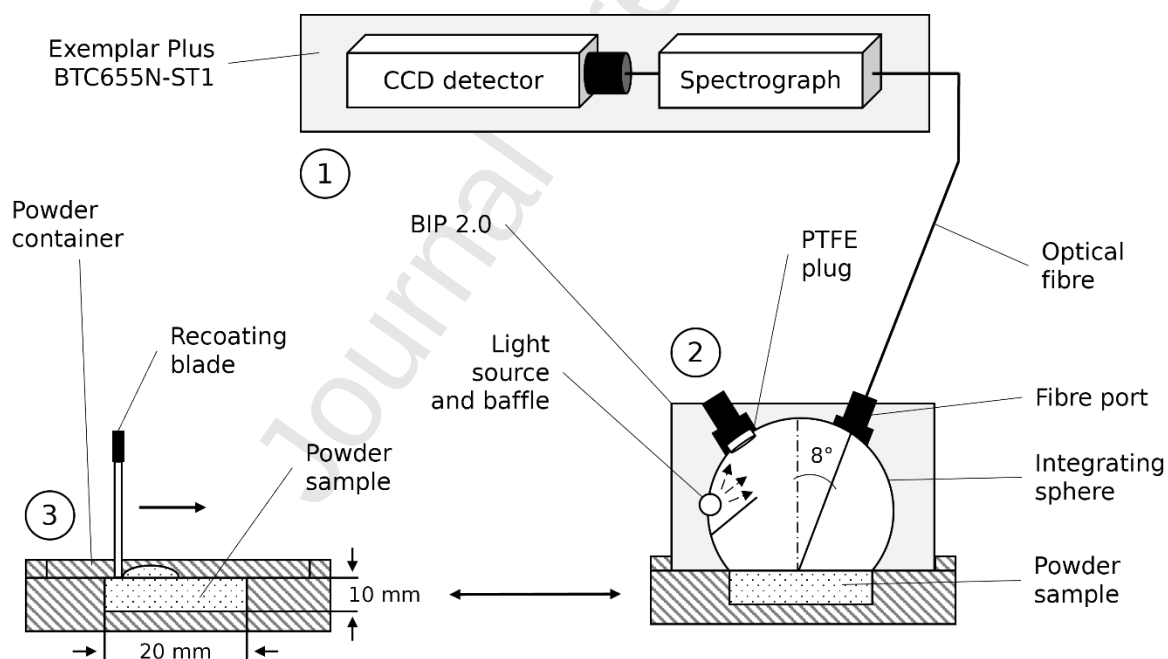


Figure 1. Schematic of the experimental DSR setup, including: 1 – powder sample container; 2 – integrating sphere set-up; 3 – spectrometer.

2.6. Data analysis

The analysis included the following single-metric properties: BD (g/ml), d_{50} (μm), VMD (μm), BFE (mJ), SI (-), FRI (-), SE (mJ/g), CBD (g/ml), slope β_0 and intercept β_1 linear fit parameters for

CI and AOR (-), and average reflectance (%). Data from these indicators were normalized using min-max scaling, where the lowest value within each set was assigned a value of 0, the highest was set to 1, and intermediate values were scaled linearly between 0 and 1. In addition, a Spearman correlation coefficient matrix was calculated to assess the monotonic relationships between the various parametric results at the powder level (separately for Al5052-based and Al5254-based powder blends). A Python script was prepared to perform the analysis, utilizing the panda's library for data manipulation and the scikit-learn library for data normalization and standardization. A Jupyter Notebook file (*SD1_data_analysis.ipynb*) containing the analysis code and a Microsoft Excel file (*SD2_data.xlsx*) containing the analysis data are included as supplementary materials.

3. Results

3.1. Morphology of Al5xxx powder blends

Figure 2 shows SEM micrographs of all the powder blends studied and corresponding EDS maps of the main additive element. In Figure 2(I–III), different morphological characteristics of the additive powders are observed. Based on these observations, ZrB₂ is identified as a dendritic powder with a highly developed surface area, while CaB₆ and Ta powders exhibit plate-like, irregular, and sharp-edged particles with a regular aspect ratio. Additionally, the particle size distribution of Ta is the narrowest, while CaB₆ and ZrB₂ contain oversized particles (see Table 1 for nominal PSD comparison of additive powders). There is a noticeable difference between the Al5052 and Al5254 base powders — the former consists of potato-shaped particles with a large number of satellite particles, while the latter is more spherical and finer, with several particles having a large surface area — as shown in Figure 2(1) and Figure 2(A), respectively. EDS maps of the main additive elements, shown as insets in the corresponding SEM images of the powder blends (Figure 2(1–6, A–F)), display the distribution of the additive powders within the base Al5xxx powders. Each powder was sampled three times at different locations within the batch volume. Within each sample, multiple spots were investigated, qualitatively showing no differences within the batch. The EDS mapping shows the presence of oversized particles in the Al5052-ZrB₂ and in all the Al5xxx-CaB₆ blends, while uniformly distributed additive particles are present in all the of the Al5xxx-Ta blends. The distribution of

specific additive particles within the base aluminum powders is similar for both Al5052 and Al5254 blends, despite the differences in the morphology of the base aluminum powders.

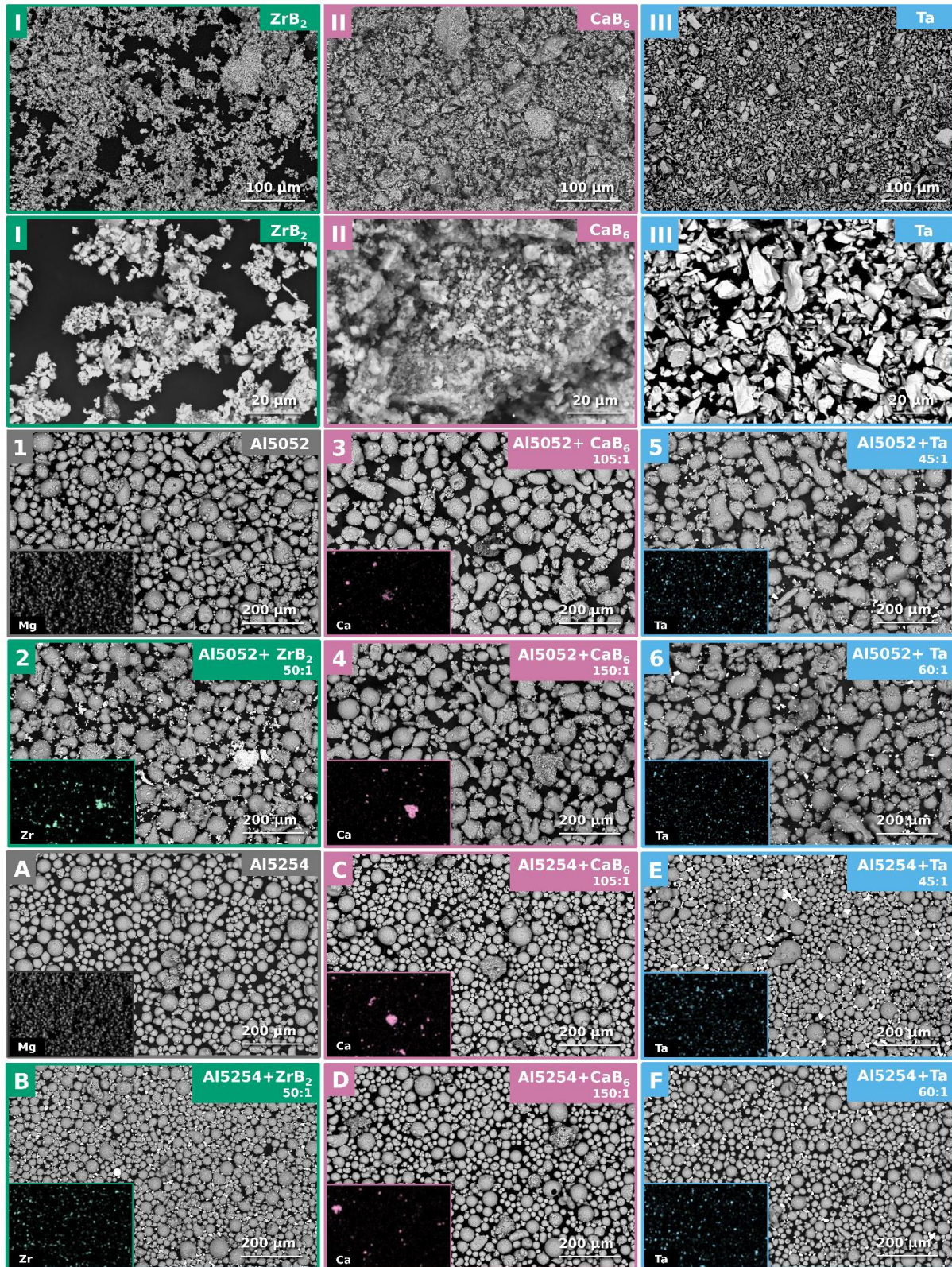


Figure 2. Micrographs of additives and Al5xxx powder blends used in the study and corresponding EDS maps of the main element of additive powder. (I) ZrB₂; (b) CaB₆; (II) Ta; (1) Al5052; (III) Al5052 +

ZrB₂ 50:1; (3) Al5052 + CaB₆ 105:1; (4) Al5052 + CaB₆ 150:1; (5) Al5052 + Ta 45:1; (6) Al5052 + Ta 60:1; (A) Al5254; (B) Al5254 + ZrB₂ 50:1; (C) Al5254 + CaB₆ 105:1; (D) Al5254 + CaB₆ 150:1; (E) Al5254 + Ta 45:1; (F) Al5254 + Ta 60:1.

Figure 3 presents the results of bulk density and PSD measurements for the studied powder blends. The bulk density of the Al5254 powder is higher than that of the Al5052 powder (Figure 3(a, d)). This is expected, as Al5254 contains a larger amount of fine and spherical particles (see Figure 2), which leads to more efficient particle packing during the container filling process for bulk density measurement, similar to what has been observed for other AM powders [25]. However, the bulk densities of different powder blends blended with the same additive powder variant (Al5xxx-additive) are nearly identical. Therefore, the bulk density is a combination of the bulk density of the base powder and the densities of the additive powders, along with the impact of the additive powder morphology on the powder packing. This observation aligns with the similar mixing effectiveness seen in the SEM micrographs, regarding additive powder loss and distribution. Thus, the blending process is unlikely to affect the experimental results discussed later in the study.

Particle size distribution measurements confirm the microscopic observations that Al5254 powder is finer than Al5052. The d₁₀–d₉₀ range for Al5254 is approximately 20–60 µm, which is close to the nominal PSD, while the d₁₀–d₉₀ range for Al5052 is above its nominal PSD, at approximately 30–80 µm. Although the additive powders exhibit visibly smaller particle sizes compared to the Al5xxx base powders, the PSD of the powder blends is shifted toward larger values in the cases of Al5052-CaB₆ 105:1 and Al5052-Ta 45:1 (Figure 3(b)), which is unexpected and affects the d₅₀ values of these powder blends as well (Figure 3(c)). SEM investigations did not reveal any changes in the powder morphology, such as agglomerates or surface modifications, that could affect the PSD as measured by laser diffraction. This suggests that the observed shift in PSD may result from processes occurring during powder feeding and spraying at 2 bar in the VIBRI dispersion unit, where the powder is dry-sprayed into the measurement window of the HELOS BR R4 + RODOS laser diffraction system. However, these processes cannot be investigated further because the powder is subsequently collected by an integrated vacuum system. Alternatively, the observed shift in PSD could also be due to measurement artifacts. For example, discrepancies exist between the PSD and powder morphology data obtained using laser diffraction and static-image analysis methods,

particularly when measuring fine and irregular powder particles [49]. Moreover, applying a spherical scattering model in laser diffraction for non-spherical particles can introduce significant artifacts. These artifacts may appear as false multimodal distributions or substantial underestimations of actual particle sizes [50]. Notably, this effect is not observed with Al5254, suggesting that the interactions between the additive particles and the base powder are likely influenced by the unique properties of the Al5254 powder (Figure 3(e, f)).

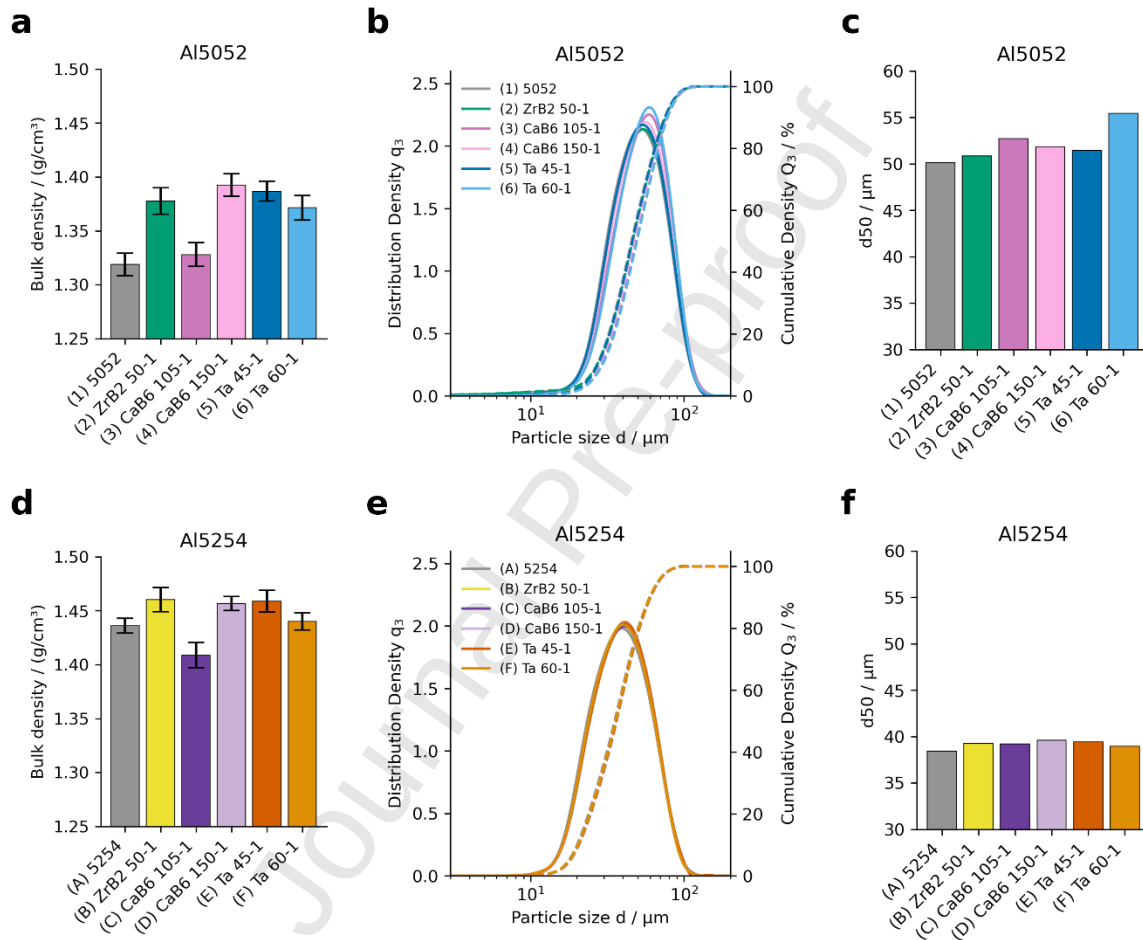


Figure 3. Bulk densities (a, d), particles size distributions (b, e) and mean particle sizes d_{50} (c, f) of Al5052-based (a–c) and Al5254-based (d–f) powder blends.

3.2. Flowability of Al5xxx powder blends

In Figure 4, the results of the rotating drum experiment are presented. A significant difference in the cohesive index values is observed between the Al5052-based and Al5254-based blends, as shown in Figure 4(a, b). The larger particle-sized Al5052 exhibits almost identical behavior across all Al5052-additive blends, similar to the base Al5052. Taking into account the confidence intervals of the measured values, the additive type affected the cohesive behavior

of the Al5254-based powder but not that of the Al5052-based group. Conversely, the base Al5254 powder only behaves similarly to the Al5254-CaB₆ 150:1 blend. The addition of Ta (at 45:1 and 60:1 ratios) increased the CI value by approximately 10 points. The influence of CaB₆ addition was less pronounced, with the CI increase in Al5254-CaB₆ 150:1 being about 5 points, which, for most drum speeds, falls within the range of confidence intervals. Generally, as the drum rotation speed increases, the CI rises, indicating shear thickening behavior. This type of flowability degradation with increasing drum speed is typically found in metal powders for AM [48], [34]. An exception is noted for the Al5254-ZrB₂ 50:1 blend, which not only lowers the CI by about 5 points at lower drum rotation speeds but also exhibits lower CI values at speeds above 30 rpm compared to the 1–20 rpm range, indicating slight shear thinning behavior. Additionally, the CI values for all Al5052 powder blends remain below or close to 30, indicating satisfactory powder flowability (CI above 30 is considered as “poor” flowability [51]). In contrast, the results for Al5254 mixed with CaB₆ 150:1 and Ta (both 45:1 and 60:1) exceed the 30 threshold, indicating that the flowability requirement is not met. Similarly, Al5052-based blends exhibit more stable AOR values across all Al5xxx-additive variants compared to Al5254-based blends (Figure 4(b, d)). In addition, all powders within each powder group exhibit similar AOR values, as the average values for most drum speeds fall within the confidence intervals. However, Al5254-based blends show a smaller increase in AOR with increasing drum speed. This is unexpected, as powders with higher flowability typically exhibit lower AORs due to reduced particle interactions, leading to smoother flow behavior [52]. The seemingly contradictory results between the CI and AOR for Al5052 and Al5254 blends highlight the complexity of powder flowability. CI suggests better flowability, while higher AOR values indicate stronger particle interactions. This discrepancy arises because CI and AOR measure different aspects of flow. CI reflects cohesiveness and resistance to flow initiation, while AOR indicates the ability to sustain flow. Powders with similar cohesion values can exhibit different flow behaviors due to variations in interparticle forces, particle size distribution, shape, and surface properties [53]. In addition, drum rotation can lead to local density variations, which can affect the observed AOR without necessarily affecting the overall flowability as measured by CI [51]. Despite higher AOR values, Al5052 blends perform better in the Hall funnel test, demonstrating that AOR alone is insufficient for assessing flowability. This is consistent with the findings of Salehi et al. [54], who observed that some powders with higher AOR may still exhibit good flow in certain practical applications

due to the influence of other factors such as particle shape and surface roughness. All Al5254 variants, including the base powder, did not flow through the funnel, while the Al5052 variants demonstrated a similar flow time, all around 30 seconds (see Table S1 in the supplementary material).

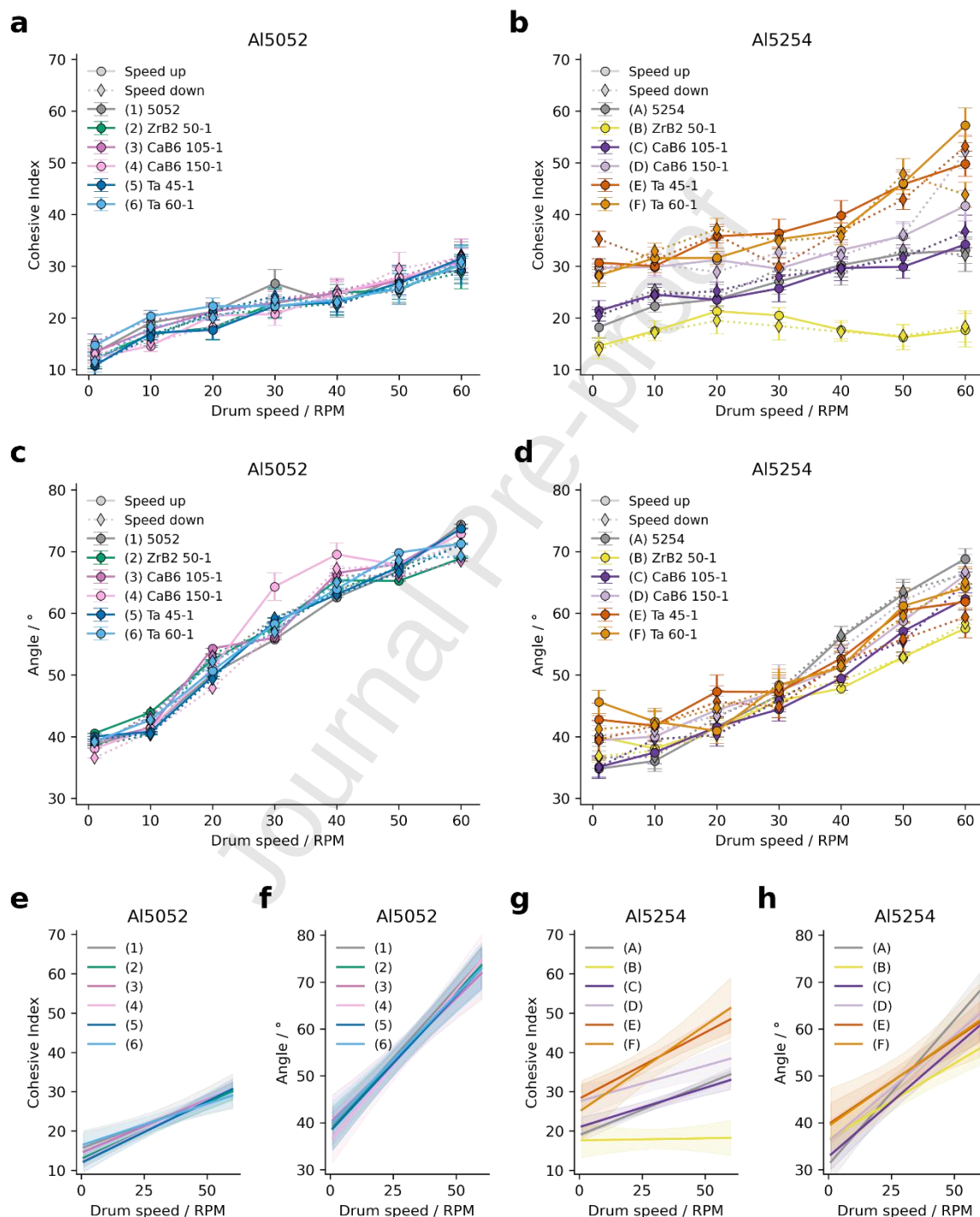


Figure 4. Results of rotating drum flowability analysis. Cohesive Index vs Drum speed (a, b); Dynamic Angle of Repose vs Drum speed (c, d); Linear regression fit of CI (e, g); Linear regression fit of AOR (f, h) for Al5052-based (a, c, e–f) and Al5254-based (b, d, g–h) powder blends. “Speed up” and “Speed

down” in the legend (a–d) indicate increasing and decreasing rotational drum speed, respectively. Errors in all plots represent confidence intervals at $\alpha=0.05$.

Analyzing both the CI and AOR graphs (Figure 4(a–d)) for both Al5xxx powder blend variants, the powders do not exhibit clear thixotropic behavior, which is typically associated with a material that does not return to its initial state after shear stress due to particle agglomeration [51]. Although there are slight differences in the CI and AOR results between increasing and decreasing drum speeds, these discrepancies are not consistent between the CI and AOR measurements. Moreover, discrepancies are observed at certain (random) speed levels, and the direction of CI changes (positive or negative) does not follow a clear pattern.

Since all powders, except the Al5254-ZrB₂ 50:1 blend, exhibit shear thickening behavior (i.e., CI increases with drum rotation speed), a linear regression model was applied to the CI and AOR data. This approach reduced the CI and AOR data from discrete points to a single value metric, characterized by the β_0 (intercept) and β_1 (slope) parameters of the linear regression model. The linear regression models, along with their confidence intervals, are presented in Figure 4(e-h). Most of the models show a good fit, with coefficients of determination (R^2) ranging from 0.74 to 0.99. This representation confirms that all 5052-based powder blends exhibit identical responses in the rotating drum experiment, rendering the powders indistinguishable based on this measurement. In contrast, the linearized data for the 5254-based powder blends enabled the separation into three groups, distinguished by increasing slopes of the regression model. Specifically, these groups include the Al5254-ZrB₂ 50:1 blend, pure 5254 along with both CaB₆-containing 5254 blends, and both Ta-containing 5254 blends.

3.3. Rheology of Al5xxx powder blends

In Figure 5, the results of the FT4 rheology test are presented. The first part of the experiment, comprising iterations 1 to 7, represents the stability test sequence, while iterations 8 to 11 correspond to the variable flow rate test sequences, as shown in Figure 5(a) for Al5052-based blends and Figure 5(c) for Al5254-based blends. During the stability test sequence, all powder blends exhibit a stability index between 0.89 and 1.2, with 10 out of 12 powders showing $0.9 < SI < 1.1$ (Figure 5(b, d)). Powders not affected by flow typically have an SI between 0.9 and 1.1 [55]. Based on the total energy plots, only one powder shows noticeable instability, i.e., Al5052-ZrB₂ 50:1 ($SI = 1.20$). This type of instability is often associated with agglomeration or

segregation during powder flow [42]. Both effects are likely to occur during testing, especially considering that ZrB_2 is a dendritic powder, while CaB_6 has plate-like, irregular particles, which can lead to agglomeration when blended with aluminum [56]. However, post-FT4 SEM investigation did not reveal any agglomeration. Representative SEM images from the post-evaluation are provided in the supplementary material (Figure S2).

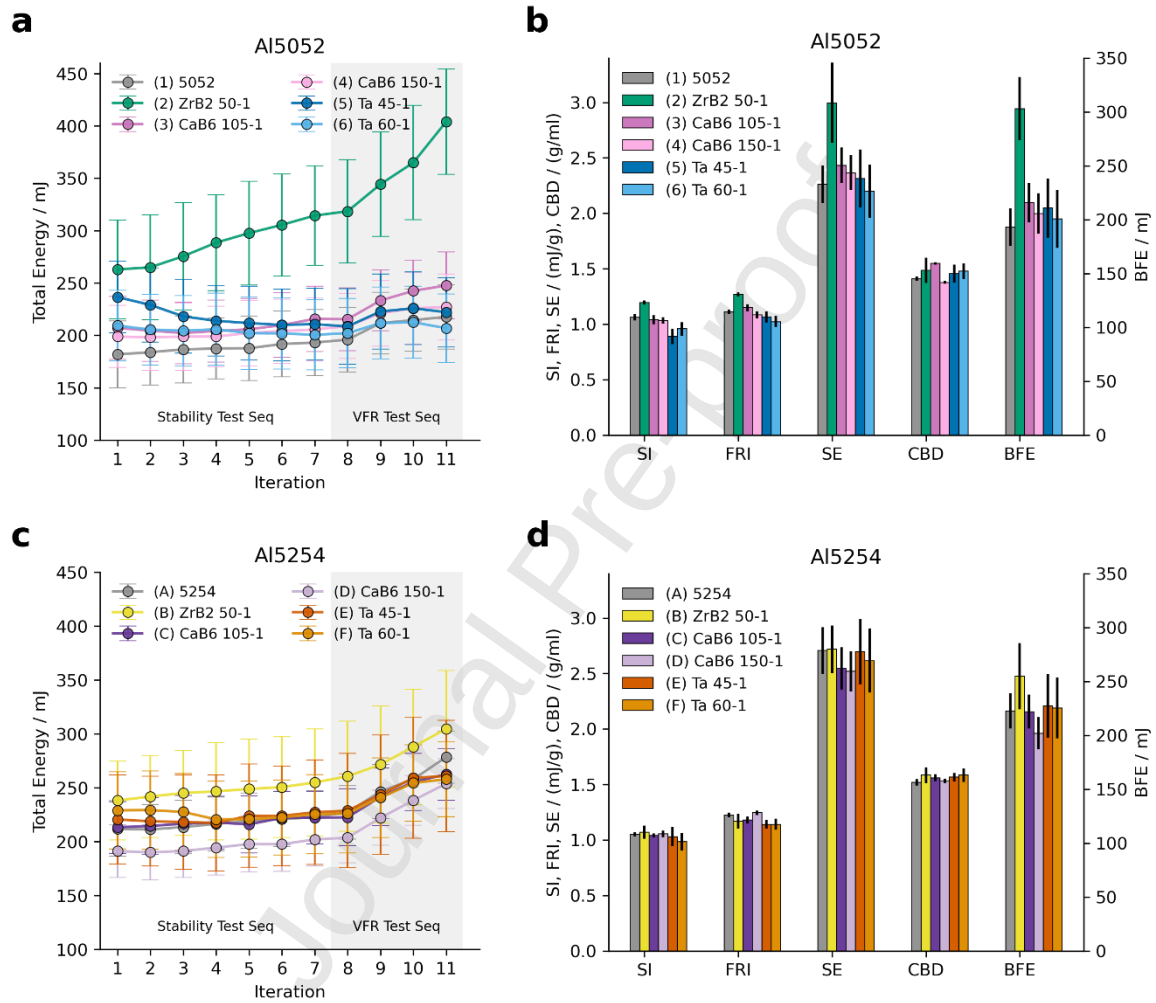


Figure 5. Results of the FT4 rheology test of Al5xxx powder blends; Total energy at subsequent test iterations (a, c), and comparative indexes derived from stability and variable flow rate sequences, i.e., BFE, SI, FRI, SE and CBD (b, d). Al5052-based blends (a, b), and Al5254-based blends (c, d).

Therefore, Al5052-ZrB₂ 50:1 instability can be associated with changes in powder flow behavior during FT4 testing. ZrB₂ powder, with particle sizes of 1–10 μm , is the smallest additive studied. Its fine and dendritic particles likely increase the Al5052-ZrB₂ blend's packing density, raising flow resistance. The small ZrB₂ particles occupy interstitial spaces between larger aluminum particles, enhancing inter-particle contacts and friction [57]. Their high

surface area-to-volume ratio amplifies van der Waals forces, which intensify as particle size decreases. Electrostatic attractions may also influence flow, especially if surface charging occurs during blending [58]. These factors, combined with the small particle size and developed surface of ZrB_2 , can strengthen inter-particle interactions, forming temporary networks during flow. Additionally, the PSD of the base powders is fairly wide, which may also contribute to the overall flow characteristics. The effect in the Al5254- ZrB_2 50:1 blend shows a similar character—BFE increases stability from the 1st to the 7th test iteration; however, due to high confidence intervals, this cannot be statistically confirmed. The effect of the other additives on powder rheology is minimal, as the registered values fall within the confidence intervals, indicating that most blends exhibit similar flow characteristics despite observed, local flow fluctuations.

Starting with iteration 8 (Figure 5(a, c)), the variable flow rate (VFR) test begins. The first iteration of the VFR test is performed with the same blade tip speed same as in iteration 7, and then the tip speed decreases. All powders show an increase in total energy starting at iteration 9. However, for Al5052-based blends (except Al5052- ZrB_2 50:1), the total energy stabilizes between iterations 9 and 11. This results in an FRI close to 1 (between 1.02 and 1.15), indicating that Al5052-based blends are almost insensitive to flow rate changes. Most powders typically fall within the range of $1.5 < \text{FRI} < 3$ [59]. Therefore, even the Al5254-based blends, which have an FRI between 1.14 and 1.25, exhibit less sensitivity to flow rate changes compared to the typical powders tested with the FT4. Similarly to SI, only the Al5052- ZrB_2 50:1 powder shows a statistically significant increase in FRI at 1.27 within both powder groups.

Based on the FT4 test sequence, additional powder characteristics were also derived (Figure 5(b, d)). The conditioned bulk density was measured at the end of the stability test, i.e., between iterations 7 and 8. The CBD values of both base Al5xxx powders were lower than those of their blends. CBD shows similar trends between the two powder types. However, the Al5052- CaB_6 105:1 powder exhibits a much higher CBD than the other Al5052-based powder blends, while Al5254- CaB_6 105:1 does not show this behavior. Another noteworthy, yet expected, result is the difference between CBD and conventional bulk density measurements. Conditioning allowed for a low-stress, homogeneous packing state [60], increasing the density of all powders by about 0.1 g/ml, except for Al5052- CaB_6 105:1, which showed a larger increase of 0.22 g/ml. Based on the confidence intervals, the differences in CBD between

powder blends within the same group are not significant. However, the difference between Al5052-based blends and Al5254 blends is approximately 0.1 g/ml, exceeding the confidence intervals, indicating that Al5254 blends have a higher CBD.

The basic flowability energy is recorded as the total energy during the last stability test (7th iteration). Generally, it is often assumed that a low BFE indicates better flow properties compared to a high BFE [61], which can be influenced by factors such as particle surface texture or the amount of additive. A comparison of the BFEs of the base aluminum powders shows that base Al5052 has a lower BFE than base Al5254 (193 mJ and 223 mJ, respectively), confirming the results from the rotating drum and funnel tests described earlier. However, considering the confidence intervals, this difference is not statistically significant. Similarly, the impact of additives on BFE varies, but the only statistically significant result is observed for Al5052-ZrB₂ 50:1, where the BFE increases by +60%, exceeding the confidence intervals. In contrast, for Al5254-ZrB₂ 50:1, the BFE increases by approximately 15%, but this result falls within the margin of error. As discussed, it is possible to be influenced by particle attrition, or changes in frictional forces between particles due to additives [60]. The observed differences can be attributed to the inherent morphological differences between the two base powders. The larger, irregular particles in the Al5052 blends are more susceptible to the influence of additives due to increased interlocking and friction, leading to higher BFEs. In contrast, the smaller and more spherical particles of the Al5254 base powder exhibit minimal changes in BFE upon the introduction of additives, as their shape reduces inter-particle friction and mechanical interlocking.

Specific energy is similar to BFE in measuring powder flow but simulates an unconfined or low-stress environment due to the upward clockwise blade motion. Unlike BFE, where compressibility plays a significant role, SE depends primarily on the shear forces between particles [62] and is normalized to the mass of the powder samples (split mass). When comparing the SE and BFE results within each Al5xxx-based powder batch, a clear correlation is observed. Among the blends, Al5052-ZrB₂ 50:1 is the only powder blend exhibiting a statistically significant increase in SE. This suggests that shear forces between particles are the main factor affecting flow in Al5xxx-based blends during the FT4 test, with compressibility playing a minimal role. The similarities between SE and BFE indicate that the flow behavior is largely related to cohesion and physical properties like particle size, shape, and surface

roughness[60]. Generally, higher SE indicates more cohesive powders [62]. All powders are characterized by $2 < SE < 2.5$, with $SE < 5$ indicating low cohesion and high flowability according to the FT4 test standard.

3.4. Reflectance of Al5xxx powder blends

In Figure 6 results of diffuse reflectance measurements are presented. During the reflectance acquisition, the powder is spread in a 20 mm diameter × 10 mm high container using a steel blade to simulate the powder bed recoating process. The container features a cutout designed to fit the blade, which is removed after the powder layering. An integrating sphere assembly, with dimensions matching the cutout, is then placed on top of the container to complete the setup. Diffuse light is directed onto the powder, and the measured reflectance represents the average reflectance from the entire spread powder area at the currently modulated wavelength (0.5 nm steps). Further details on the assembly and setup are provided in reference [33] and the supplementary materials (Figure S2). Figure 6(a, c) show the reflectance across the entire measured spectrum (900–1100 nm), while Figure 6(b, d) display the average reflectance within this spectrum. For both Al5xxx variants, the reflectance is highest for the pure aluminum powders. Despite their similar chemical composition, the average reflectance difference between pure Al5052 and Al5254 powders is approximately 3 percentage points. This difference is attributed to variations in morphology and particle size distribution between the powders, as reflectance measurements are sensitive to these factors due to light scattering and eventual light trapping. Powders that promote higher light trapping naturally absorb more light [63]. In this case, the Al5052 powder blends exhibit a lower bulk density and larger particles with more complex shapes, which offer greater surface area for light scattering and trapping, resulting in lower reflectance.

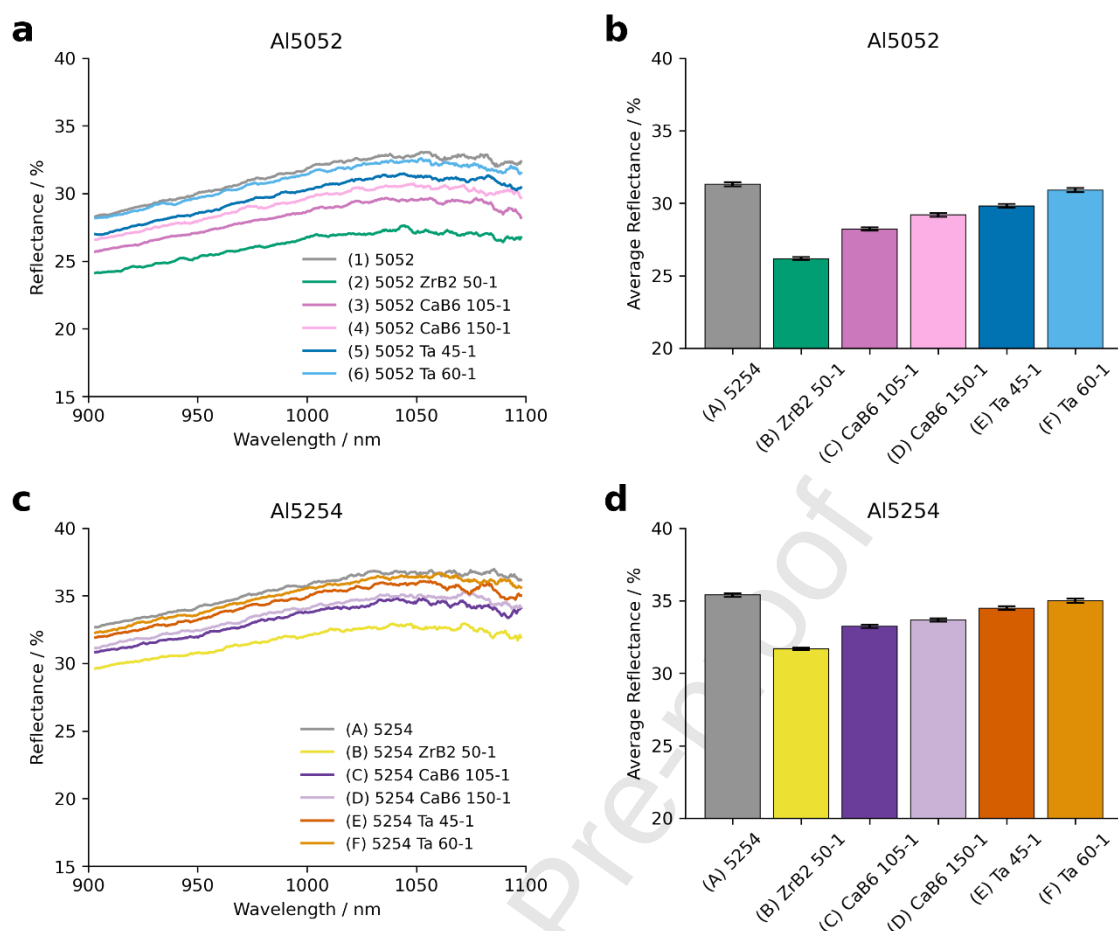


Figure 6. Diffuse reflectance of Al5xxx powder blends. Full acquired spectrum smoothed by 10 point moving average (a, c) and average reflectance of the full spectrum (b, d); Al5052-based blends (a, b), and Al5254-based blends (c, d).

All the additives decrease reflectance for both powder variants, and the relationship between the additive type and the decrease in reflectance is consistent across both Al5xxx powders. The addition of ZrB₂ results in the largest decrease in reflectance, followed by CaB₆ 105:1, CaB₆ 150:1, Ta 45:1, and Ta 60:1. Higher amounts of additive result in greater reflectance drops, which can be observed for both CaB₆ and Ta additives. Aluminum is a highly reflective material, with high reflectance throughout the visible and infrared spectrum, including the near-infrared (NIR) spectrum used in the DRS experiment. Although ceramics such as ZrB₂ also have high NIR reflectance [64], [65], their addition to aluminum powder can still reduce the overall diffuse reflectance due to factors like increased scattering, absorption of scattered light, and interactions at particle interfaces [63]. This is in consistent with the observed relationships, as ZrB₂ produces the largest reflectance drop when added at the highest weight fraction, while CaB₆ at a higher wt.% addition (105:1) produces a smaller drop than at a lower wt.% addition

(150:1). With Ta addition, the relationship is simpler. Al generally has a higher reflectance than Ta, especially in the visible and NIR ranges. This is because Al has both a relatively high refractive index and a lower extinction coefficient (less absorption), leading to more light being reflected [66]. Therefore, the addition of another metallic powder simply reduces the overall reflectance of the powder blend due to higher Ta absorption.

The only considerable difference in the powders' response to DRS is the spread of the measured reflectance values. Between pure Al5052 powder and the Al5052-ZrB₂ blend, a spread of 5 percentage points is measured, while for pure Al5254 powder and the Al5254-ZrB₂ blend, the spread is 3 percentage points. This difference is likely due to the powder morphology and the way diffuse light interacts with trapped particles. As discussed, Al5052-based powder blends are characterized by larger and more irregularly shaped particles, which result in lower bulk density (see Figure 3(a, d)). This creates more gaps between particles, potentially increasing the exposure of additive particles and leading to greater variations in reflectance, as well as an overall lower reflectance in Al5052-based powder blends.

4. Discussion

The preparation of powder blends is essential for developing alloys used in additive manufacturing. However, preparing these blends can be challenging, with results varying based on several factors, such as particle size distribution, the morphology of both the base and additive powders, blending methods, and other factors. Inconsistent powder properties, such as poor particle dispersion or non-uniform additive distribution, can lead to heterogeneous microstructures [19], conglomerate formation [18], and unpredictable mechanical properties of the fabricated parts [21]. Additionally, the blending process can result in the loss of additive concentration, further impacting the quality of the blend [22]. Current assessments of powder blends for AM are often qualitative, relying heavily on SEM investigations, as demonstrated in referenced works focusing on the material properties achievable from such blends. However, these qualitative approaches are insufficient for providing standardized, reproducible metrics necessary to ensure consistency and reliability in AM processes.

In this study, we aimed to identify a reliable metric for evaluating powder blends for AM. To ensure blend homogeneity, we implemented a sampling and blending protocol involving

small-batch blending (~100 g) for each powder. The homogeneity of each blend was verified using SEM analysis at three different sample locations for each powder batch, with no variations detected within the batches. The measurement techniques used provided several single-value metrics describing various powder characteristics, including bulk density, d_{50} , VMD, BFE, SI, FRI, SE, CBD, β_0 and β_1 parameters of the linear regression model for CI and AOR, as well as average reflectance. The objective was to identify a metric that demonstrated consistent behavior for both AI5052-based and AI5254-based powder blends, with the future aim of scaling the approach to large-scale blending scenarios involving significantly larger volumes.

To facilitate clear comparisons between the acquired metrics, we applied min-max normalization, adjusting each metric so that its minimum value was 0 and its maximum value was 1. The normalized values were then compared between the AI5052-(x) and AI5254-(x) powder blends, where x denotes the same additive type and weight fraction. Normalization reduced the impact of differences in powder characteristics, such as PSD and bulk density, enabling the identification of consistent trends. For instance, powders with larger PSDs exhibited lower cohesion indexes in rotating drum experiments compared to those with smaller PSDs [34]. By normalizing the data, we highlight clearer and more consistent correlations between powders, independent of absolute values or inherent variability. The resulting normalized difference matrix is presented in Figure 7(a), while the mean normalized difference for each metric across all powder blend pairs is shown in Figure 7(b).

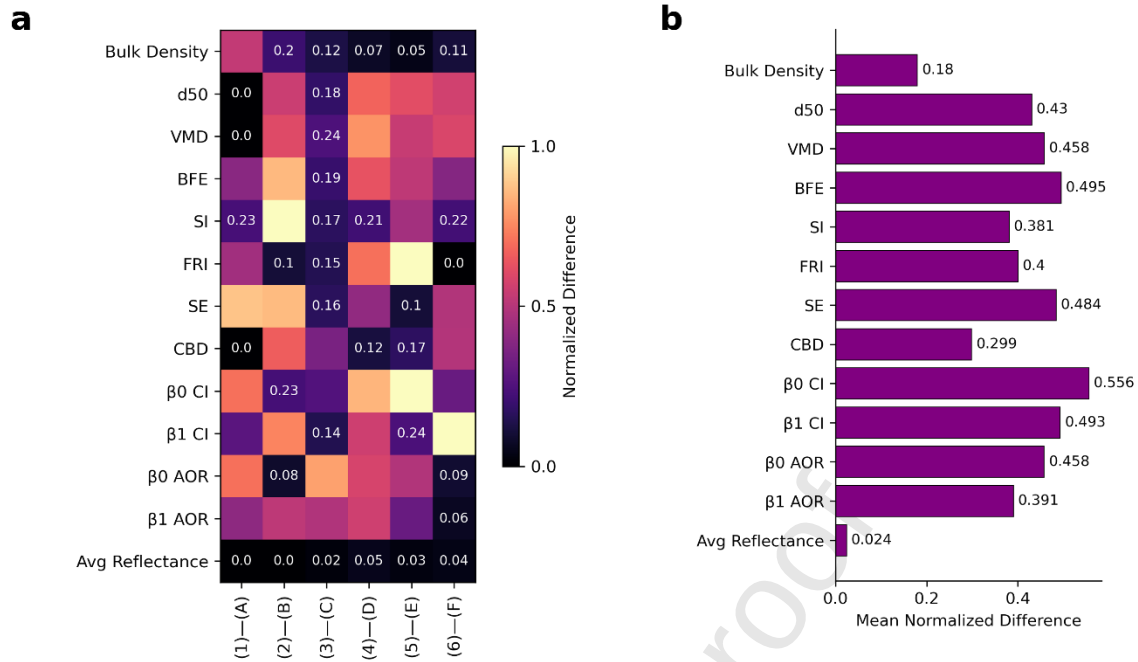


Figure 7. Comparison between Al5052-based and Al5254-based powder blend pairs; (a) Normalized difference matrix—only normalized difference values below 0.25 are displayed. Pairs on the x-axis are labeled as shown in Table 1; (b) Mean normalized difference across all the pairs.

Two metrics clearly stand out — bulk density and, particularly, average reflectance. The mean normalized difference in bulk density between Al5052-(x) and Al5254-(x) variants is 0.18, while for average reflectance, it is as low as 0.024. This indicates that each additive powder produces nearly identical normalized changes in reflectance for both Al5052-based and Al5254-based blends. As discussed earlier, Al5052 blends exhibited lower bulk density and larger, more complex particles than Al5254, resulting in greater light scattering, which leads to deeper penetration and increased interaction with additive particles. The average reflectance likely remains consistent due to its sensitivity to factors such as the morphology and chemical composition of the investigated surfaces [67]. Since both Al5052 and Al5254 powders are aluminum alloys with similar surface properties, the optical behavior in terms of light reflection remains comparable. The additives that uniformly blends with the base powders, resulting in similar changes in reflectance — averaged over the entire measured powder layer — regardless of the base powders' slight morphological differences. Moreover, the minimal mean normalized difference in average reflectance (0.024) between the Al5052-(x) and Al5254-(x) blends indicates that differences in PSD and morphology between the base powders can be decoupled from the DRS measurement. Consequently, DRS reliably detects the presence and concentration of additives regardless of inherent variations in base powder

characteristics, such as PSD and particle morphology. Therefore, DRS can effectively capture and quantify both the type and ratio of additives in aluminum powder blends.

It is also observed that for the Al5052-CaB₆ 105:1 and Al5254-CaB₆ 105:1 pair, the normalized values are below 0.25 for most metrics, except CBD and slope/intercept parameters of linear fit model for CI and AOR data. However, this trend is not seen in the Al5052-CaB₆ 150:1 and Al5254-CaB₆ 150:1 pair. The absence of a consistent trend in the Al5052-CaB₆ 150:1 and Al5254-CaB₆ 150:1 pair suggests that the higher addition of CaB₆ may significantly alter the FT4 flow behavior. One possible explanation is that the increased amount of CaB₆ particles leads to enhanced particle-particle interactions due to their irregular shape and higher surface area, which can increase the powder's cohesiveness and resistance to flow [68]. The increased cohesiveness may affect the flow energy measured by the FT4 rheometer, causing discrepancies between the powder blends, as agglomeration—another potential factor—was not confirmed in the post-FT4 SEM evaluation. Therefore, the same additive may affect the flow properties differently in the two base powders due to their morphological differences.

Given these observations, we employed the Spearman rank correlation to analyze the data further. The Spearman rank correlation is effective when the data is ordinal, when the relationship is non-linear but monotonic, or when the data distribution is skewed or has outliers. The correlations between each single-value metric within each Al5xxx-based powder blend group using the Spearman rank correlation coefficient matrix, are shown in Figure 8(a) for the Al5052-based group and in Figure 8(b) for Al5254-based group. This analysis aimed to assess whether different test results were correlated within a single Al5xxx-based powder blend group. A Spearman rank of $\geq |\pm 0.4|$ indicates a moderate relationship, $\geq |\pm 0.6|$ indicates a strong relationship, and $\geq |\pm 0.8|$ indicates a very strong relationship.

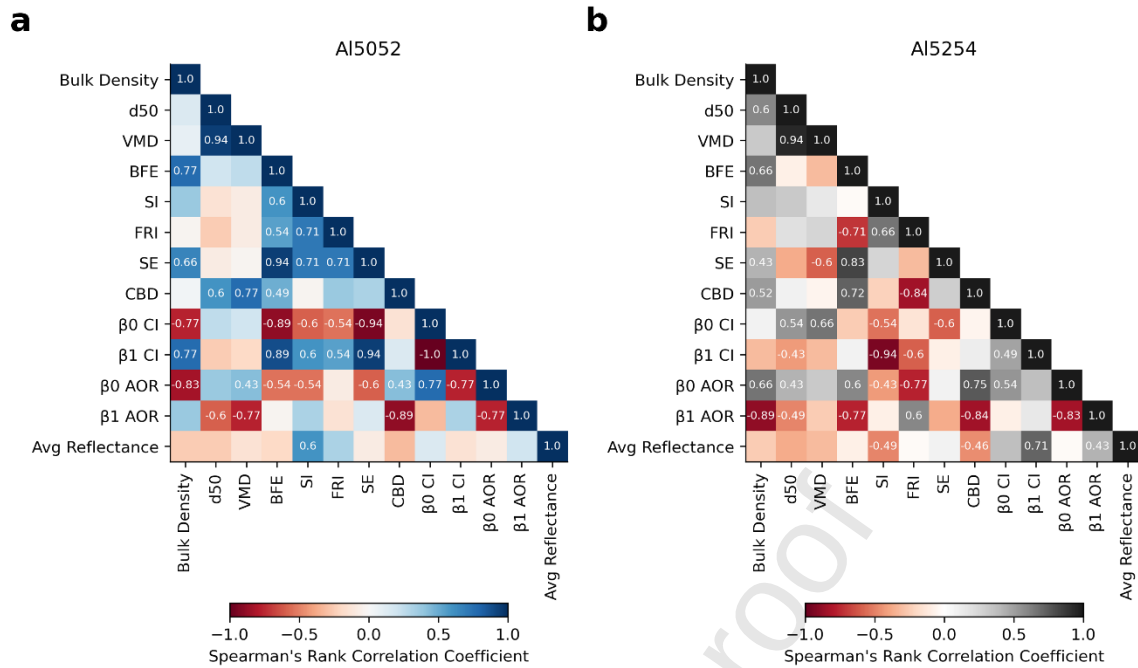


Figure 8. Spearman correlation coefficient matrix between each single-value metric—Spearman rank values are only shown if moderate or higher ordinal relationship ($\geq |\pm 0.4|$) is calculated; (a) Al5052-based powder blends; (b) and Al5254-based powder blends.

The strong correlations (Spearman rank $\geq |\pm 0.8|$) between a significant number of FT4 parameters (BFE, SI, FRI, and SE) and the linear fit parameters of CI and AOR rotating drum data in both powder types suggest that these metrics are linked through the powders' cohesive properties. Thus, both tests capture the influence of additive powders on cohesive behavior and flowability in a similar fashion. However, spearman ranks also indicate that Al5052-based and Al5254-based powder blends show different correlations between the captured metrics. Such behavior can be attributed to the differing influences of the additive powders on flowability and rheology between the two powder groups, as well as the morphology of the base powders themselves. The Al5254 powder, with smaller and more spherical particles, tends to pack more efficiently due to its spherical shape [69]. However, smaller particles typically exhibit lower flowability due to increased cohesive forces resulting from a higher surface area to volume ratio [70]. In contrast, the larger, less spherical particles of Al5052 have higher surface roughness and irregular shapes, leading to increased interparticle friction and mechanical interlocking [71]. These morphological differences lead to a higher angle of repose and lower cohesion for pure Al5052 compared to pure Al5254 (see Figure 4(a-d)). Looking at the CI and AOR plots, additives did not significantly change the flow

behavior of Al5052-based blends, contrary to Al5254-based blends. This means that additives may disrupt the packing structure more significantly for fine and spherical Al5254-based blends, thereby altering the flow behavior. In Al5052-based blends, additives may fill these voids or adhere differently to the particle surfaces, resulting in different changes in flowability and rheology. The larger contact areas and higher asperity interactions in Al5052 powders can dampen the effect of additives on flow properties. Ultimately, while the rotating drum and FT4 tests produce comparable results, they fail to account for the fact that, despite differences in morphology, the base powders are blended with the same types and amounts of additives. When DRS is applied over a powder layer, it captures averaged optical properties that correlate with the morphological and chemical properties of the measured powder. DSR can detect, for instance, surface roughness, oxidation, and particle size distribution changes. Oxidative processes can alter the surface structure of materials, introducing new absorption bands or shifting existing ones in the reflectance spectra [72]. The size and distribution of the particles influence the path length of light within the powder layer [63]. Thus, DRS can provide information on the powders' PSD, morphology and surface condition.

Our previous studies demonstrated that DRS measurements are a non-destructive and efficient method for monitoring key powder characteristics such as oxidation, PSD, chemical composition, and powder homogeneity — critical factors for quality control in AM. Specifically, we showed that DRS can provide quantitative information about powder oxidation and PSD changes through simple regression models, validated with various nickel- and titanium-based alloy powders [33]. For example, we established a strong correlation between DRS and mass spectrometry results for intentionally oxidized powders, achieving a log-linear fit with an R^2 coefficient of up to 0.99 and demonstrate the capability of DRS for oxidation monitoring in metal powders.

This capability was further demonstrated in Inconel 718 and AlSi7Mg0.6 powders, where DRS identified changes in powder conditions and successfully distinguished between virgin and oversized, oxidized spatter powders [34], [48]. Beyond PSD and oxidation, DRS was useful in detecting compositional modifications, such as identifying differences in Inconel 718 powders modified with micron-sized carbide additives [2]. The reduced reflectance caused by carbide addition correlated with improved density in PBF-fabricated samples, confirming the relationship between compositional changes and processing outcomes.

Our findings extend the body of work by demonstrating that DRS is effective in detecting the presence of powder additives in Al5xxx-based powder blends. Given the discussed results of min-max normalization, there was only a 2.4% difference in average reflectance between Al5052-additive and Al5254-additive pairs. This indicates that DRS reliably detects additives and weight ratios, with reflectance changes driven by additive properties rather than base powder morphology — unlike other tested methods.

Furthermore, we propose adapting DRS for in situ application within a PBF machine to monitor powder bed homogeneity across an entire layer. By sampling multiple points, DRS could provide real-time data on blend uniformity for proactive quality control. Similar to near-infrared spectroscopy in pharmaceutical processes [73], which monitors blend uniformity through reflectance data, DRS could achieve similar results in AM. These capabilities could extend to oxidation, PSD, and chemical composition monitoring. Incorporating data-driven approaches, such as machine learning, could further enhance DRS by enabling real-time defect detection, predictive modeling, and process optimization.

These findings position DRS as a versatile, precise, and non-destructive analytical tool for AM powder quality control. Its demonstrated effectiveness in oxidation detection, PSD monitoring, chemical composition analysis, and powder bed homogeneity assessment makes it an invaluable technique for improving AM processes like PBF. Future research should focus on further refining the DRS method, particularly for in situ applications, and integrating data-driven approaches, such as machine learning, to enhance its predictive capabilities.

5. Conclusions

Two sets of Al5xxx-based powder blends, intended for powder bed fusion additive manufacturing, were mixed with ZrB_2 , CaB_6 , and Ta and evaluated for morphology, flowability, and diffuse reflectance. Several powder characterization techniques were employed, including SEM imaging, PSD analysis, bulk density measurements, flowability assessments using rotating drum and Hall funnel tests, and FT4 rheology tests, to assess both compositional homogeneity and powder layer cohesion—factors closely tied to process control. Diffuse reflectance spectroscopy was also utilized to measure diffuse reflectance and explore its potential for assessing powder blend layer homogeneity. The following conclusions were drawn:

- SEM imaging and EDS mapping revealed that the distribution of additive particles (ZrB_2 , CaB_6 , and Ta) within the base aluminum powders was similar for both Al5052-based and Al5254-based blends, despite differences in base powder morphology. This indicates that the blending process effectively distributed the additives throughout the base aluminum powders, allowing for meaningful comparisons.
- In both Al5052-based and Al5254-based powder blends, the linear regression model of the cohesive index and dynamic angle of repose derived from the rotating drum experiment showed strong Spearman rank correlations with more than half of the FT4 metrics. However, the addition of additive powders (ZrB_2 , CaB_6 , and Ta) affects the rotating drum and FT4 results without statistical significance, likely due to the high instability of powder behavior during the experiments.
- The ZrB_2 additive exhibited a statistically significant influence only in specific cases: the Al5052- ZrB_2 50:1 blend in the FT4 experiment and the Al5254- ZrB_2 50:1 blend in the rotating drum experiment. This is attributed to ZrB_2 's fine particle size and dendritic-like shape, which result in a high surface area-to-volume ratio and amplified inter-particle forces.
- Notably, ZrB_2 affected flowability and rheology differently across the experiments. In the rotating drum experiment, cohesion decreased for the Al5254- ZrB_2 50:1 blend compared to pure Al5254. Conversely, in the FT4 experiment, the Al5052- ZrB_2 50:1 blend showed increased metrics such as BFE and Total Energy compared to pure Al5052, indicating greater resistance to flow.
- The Al5052 base powder has potato-shaped particles with many satellites, while Al5254 is finer, more spherical, with larger surface areas. Min-max normalization and a normalized difference matrix revealed that these morphological differences affect the rotating drum and FT4 experiments differently, leading to a lack of strong correlation between the results.
- The additive powders ZrB_2 , CaB_6 , and Ta produced nearly identical normalized changes in reflectance for both Al5052-based and Al5254-based powder blends, with a minimal 2.4% difference in average reflectance between the two. This consistency highlights the effectiveness of diffuse reflectance spectroscopy in detecting additive type and weight ratios, as its sensitivity to surface characteristics remains unaffected by variations in base aluminum powder morphology.

- We propose adapting DRS for monitoring of powder bed homogeneity in PBF machines, providing real-time blend uniformity data for quality control. Its capabilities could extend to defect detection, predictive modeling, and process optimization, further enhancing its role in additive manufacturing layer control.

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Declaration of Competing Interest

Authors declare no competing interests.

Data Availability

Data will be made available on request.

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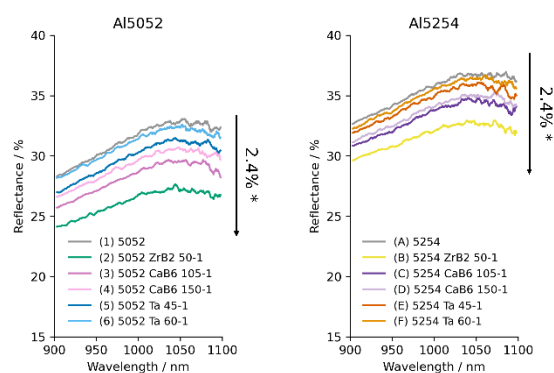
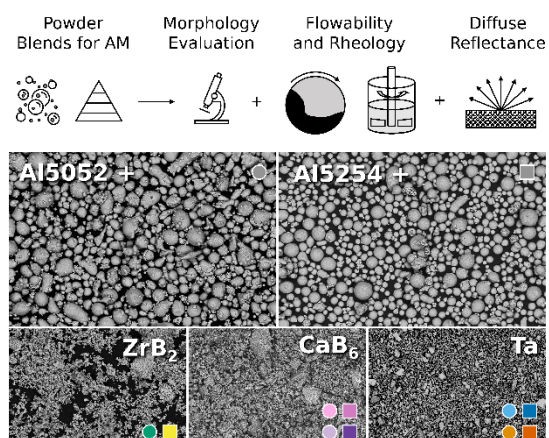
Journal Pre-proof

Declaration of interests

☒ The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

☐ The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

Graphical abstract



* Normalized difference in average diffuse reflectance between corresponding Al5052-additive and Al5254-additive pairs

Highlights:

- Al₅xxx powders are blended with varying proportions of ZrB₂, CaB₆ and Ta
- Powder morphology, flowability, rheology and diffuse reflectance are evaluated
- Flowability and rheology metrics are erratic across powder-additive combinations
- Diffuse reflectance spectroscopy (DRS) reliably detected additive type and ratio
- DRS is recognized as a potential tool for powder bed homogeneity quality control