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Rheology of Powders: Assessing the Robustness and Impact of Humidity, Tribocharging, Particle Size and Composition.

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Abstract

Many powder metallurgy (PM) processes rely on powder flowability to ensure the productivity and stability of manufacturing process and the quality of the final parts. Different methods have been developed to quantify powder flow behavior under different conditions (Hall flow, Carney flow, angle of repose, powder rheometer, avalanche, etc.). Powder rheology, which measures the resistance seen by an impeller when moving through a cylinder filled with powder, has recently generated interest in the PM community. In order to use the method for quality control, certification, simulation and R&D purposes, it is important to evaluate the robustness of the method and investigate the effect of experimental conditions and particle characteristics on the measurements. This paper presents an assessment of the variability of the results using a commercial rheometer and an evaluation of the impact of humidity, measurement vessel, charging and powder characteristics (size distribution and composition) on the measurements. Results were compared with values obtained using Hall and Carney flowmeters.

Introduction

The flowability of powder has an important impact on various manufacturing processes, including additive manufacturing (AM). In powder bed machines, the flowability impacts the ability to efficiently spread a thin and dense layer of powder without defects. Flowability affects the powder layer characteristics (density, uniformity, thermal conductivity) which influence the formation of the melt pool, defects, part performances and process reliability [1,2,3,4,5].

Flowability is not an intrinsic property of a powder but represents its behaviour when exposed to deformation. Flowability is affected by powder characteristics (particle size distribution, sphericity, presence of satellites, density, surface topologies, etc.), environmental and handling conditions (humidity, temperature, atmosphere and measurement method) as well as the flow conditions (plastic, inertial, fluidized and entrained flow) [6]. Different techniques have been developed (e.g. Hall, Carney, Gustavsson and Arnold flowmeters, shear tests, Flodex, static and dynamic angle of repose) to quantify the flow behaviour that needs to be quantified. Even if most of the tests appear to be relatively simple, important variations in the results have been reported. For example, the American Society for Testing and

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Materials (ASTM) standard B213-17 [7] reports variabilities as high as 21 % for Hall flow measurements acquired in different laboratories. Results obtained in an interlaboratory study coordinated by ASTM B09 with 25 different laboratories showed a large span and relative standard variation in Hall flow results ($Hall\ flow\ rate_{average} = 32.4\ \pm 5.9\ sec$)) [8]. A study conducted on the rheology of AM Ti6Al4V powder showed that significant variations were observed inter and intralaboratories [9]. While efforts were done to improve the repeatability, it was not possible to minimise these variations or define their sources. These variations might be problematic if the results are used to control a process or for certification.

The American National Standards Institute (ANSI) and America Makes reported [10] that the existing standards for flowability measurements do not account for the range of conditions that a powder may encounter in AM processes. Users of AM machines have reported that powders appearing to be identical when characterized using standard methods may behave differently in the machines. There is thus a need to improve, adapt or develop methods to quantify flowability more accurately and representative of the conditions encountered in AM process.

New techniques have been recently developed to evaluate the rheology of powders [11,12]. It is, however, important to understand how the powder properties and testing methods affect the results and assess the robustness of these methods to support powder qualification, quality control, certification, simulation, R&D and product development.

The objective of the work presented in this paper was to evaluate the effect of measurement conditions and powder properties on the flow behavior for different powders. The flowability tests were conducted with funnel flowmeters (Hall and Carney) and a powder rheometer (Freeman FT4) on different powders; titanium (commercially pure and Ti6Al4V), stainless steel (316L) and aluminum (commercially pure and A357) powders with different particle size distributions. The influence of the material of the vessel, humidity, operators, handling and static charges on the flowability was investigated. Different flow indexes were compared and correlated with powder properties such as the particle size distribution, morphology and composition.

Experimental Procedures

Tests were conducted with the powders presented in Table 1. Scanning electron micrographs of the aluminum (CpAl and A357) powders were taken at different magnifications using a Hitachi S-4700 scanning electron microscope. Observations were made directly on the powder but also on polished cross sections.

Table 1: Description of the powders characterized in this study.

Powder	Size distribution	Manufacturer
Commercially pure titanium (CpTi)	0-45 µm	AP&C
Ti6Al4V	0-45 µm	AP&C
316L	32-66 µm	Sandvik Osprey
Commercially pure aluminum (CpAl)	Various	Equisphere
A357	Various	Equisphere

The shape of the particles was evaluated by image analysis on polished cross sections (SEM, 250x) using a Clemex image analyzer (Clemex Vision Professional Edition, version 8.0). Sphericity was calculated using the following equation:

$$Sphericity = \frac{4\pi Area}{Perimeter^2} \quad (1)$$

The ratio of the inner diameter (ID) over the outer diameter (OD) was also used to assess deviations form sphericity:

$$r_s = \frac{ID}{OD} \quad (2)$$

where ID and OD are respectively defined as the smallest and greatest diameter of the circle centered on the centroid of the particle whose edge is the closest and farthest to the particle centroid. The centroid is defined as the center of mass of each particle. As the measurements were conducted on polished cross sections, the average values were dependent on the size distribution but also on the depth from which the particles were sectioned.

Hall and Carney flow tests were performed by measuring the time required for 50 grams of powder to flow through the funnels. Flow energy, with and without aeration, was measured using an FT4- Freeman powder rheometer (Tewkesbury, UK). Flow tests (stability, variable flow and aeration) were evaluated by measuring the energy required to move a rotating stainless-steel impeller (23.5 mm diameter) inside a glass cylinder (25 mm diameter) filled with powder, calculated from the torque and linear force meters [13]. Rheology tests were performed after conducting the conditioning cycle recommended by the manufacturer. The conditioning cycle consists of gently disturbing the powder using the impeller to generate a test sample with a reproducible packing and consolidation.

Stability tests were performed by measuring the flow energy of the impeller as it moved downward, forcing the powder towards the bottom of the cylinder. The speed of the impeller tip was fixed at 100 mm/s for the first 8 tests. Seven repeat tests were conducted on each sample with a conditioning cycle between the tests. The basic flowability energy (BFE) index represents the energy measured at test repeat #7, as set in the FT4 procedure. At the end of each stability test, the velocity of the impeller was varied from 100 mm/s down to 10 mm/s.

Specific energy (SE) was measured as the impeller moved upward, where the flow is under unconfined conditions. SE was calculated by dividing the flow energy (average of repeat #6 and #7) by the apparent density of the powder (measured with the graduated cylinder of the instrument and a balance).

The sensitivity of the flowability to aeration and air flowing through the powder was measured using aeration tests. Aeration tests were performed by measuring the flow energy as a function of the velocity of dry air (from a bottle) introduced through a porous mesh at the base of the cylinder. The air velocity was varied from 0 to 20 mm/s, with a conditioning before each test cycle. The normalised aeration sensitivity (NAS) was calculated by dividing the change in energy to move the impeller over the change in pressure during the test:

$$\text{NAS} = \frac{\Delta Energy}{\Delta Air\ flow} \quad (3)$$

Charges of the powder were measured using a surface DC Volmeter (AlphaLab, Model USSVM2, Range at 2.54 mm: 0 ± 20 V, Resolution: 0.1 V, Accuracy: ± 2% of the reading) at a distance of 2.54 mm from the top of the powder within the rheometer cylinder. The meter measures the magnitude and polarity of static charge on the surfaces of objects.

Results and discussions

Effect of the material of the vessel

Figure 1 presents the BFE of commercially pure titanium (CpTi) and Ti6Al4V alloy powders measured using the rheometer and different vessel materials (borosilicate glass, stainless steel and aluminum). The results indicate that the BFE significantly depends on the material of the vessel used. This indicates that the flow stresses measured with the rheometer are not only related to interparticle friction but are also affected by the interaction of the particles with the surface of the vessel. This is a consequence of the configuration of the test set-up, including the fact that the gap between the tip of the impeller and the vessel wall is only 750 µm. Therefore, the friction of the powder between the impeller and the vessel represents a significant portion of the measured stresses. This contribution should be monitored and remain constant as the vessel wears out or is replaced. This could be done using 'reference' materials, but the methodology needs to be developed and validated. It is important to specify that the effect of the interaction with the surface of the meters has been observed with other methods (e.g.: Hall and Carney meters).

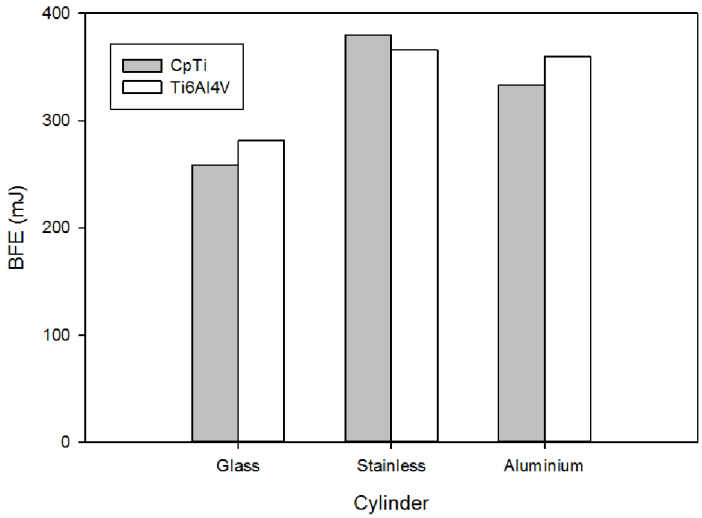

Figure 1: Effect of the material of the vessel on BFE.

Effect of humidity

For CpTi and Ti6Al4V, humidity has a limited impact on the BFE (Figure 2a), while the effect on SE is significant (Figure 2b). The differing behaviours observed for BFE and SE originates essentially from different stresses imposed during the tests. The BFE index is measured in a confined flow configuration where the impeller is moving downward, pushing the powder towards the bottom of the cylinder which generates compressive forces. The BFE index is thus affected by mechanical interaction (i.e. interlocking and surface roughness) between the particles but is less sensitive to weak interaction forces between the particles. As the impeller moves upward during the measurement of the SE, the powder is under unconfined flow. This condition creates lower consolidation forces and the measurements are more sensitive to weak interaction forces between the particles, such as those caused by humidity.

Figure 2b suggests that the powder is adsorbing humidity during the tests (or during the preparation of the tests) and affects the results. This observation indicates that the adsorption occurs quickly as the samples were tested immediately after collecting the powder from the bottle. If not controlled, the adsorption of humidity may be a potential source of variability of the results. Therefore, humidity should be monitored when analysing SE results, especially if the powder is hygroscopic.

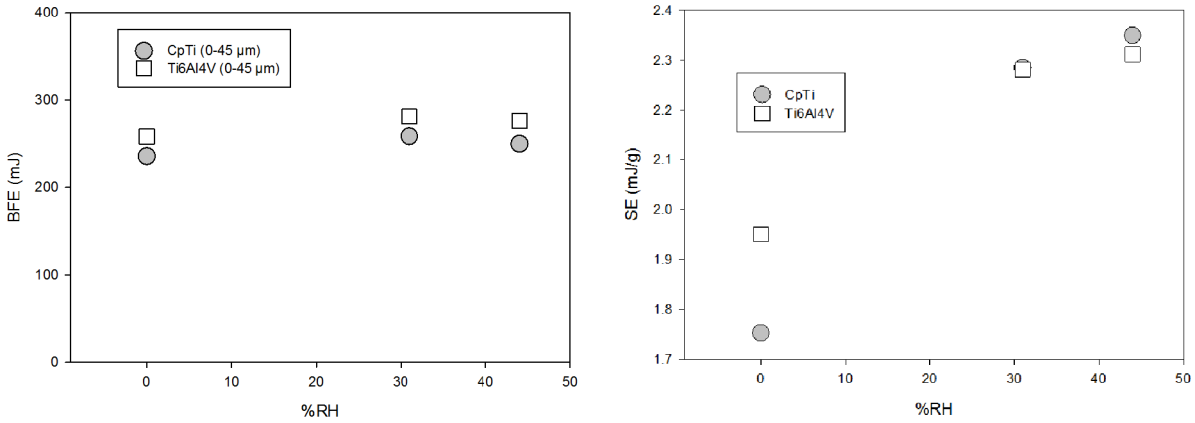

Figure 2: Effect of humidity on a) BFE and b) SE indexes.

Repeatability and effect of tribocharging

Tests were conducted to understand the sources of variations observed in an interlaboratory study [9] and to evaluate the effect of operators on the variability of the results. Dynamic tests were carried out by four operators the same day using the same conditions (i.e. laboratory, FT4 equipment and procedure). The same powder sample was used for all tests and poured back in a plastic dish between each successive test. At the end of this set of measurements with the four operators, the first operator repeated the test again using the same procedure. Figure 3a shows that the BFE values are decreasing with each repeat. It was concluded that this behaviour was not caused by the operator but by a modification of the powder properties during testing and handling. To confirm this hypothesis, the tests were repeated by an operator with the same procedure (i.e. the powder sample was poured back in a plastic dish between each successive test). The results presented in Figure 3b show that the BFE values still decrease with each repeat when measured by the same operator. Similar tests were conducted with an aluminium cup to verify if the material of the dish or cup has an effect on the BFE variations. The trend observed (not shown here) was identical and a reduction of the BFE values was observed after each successive test.

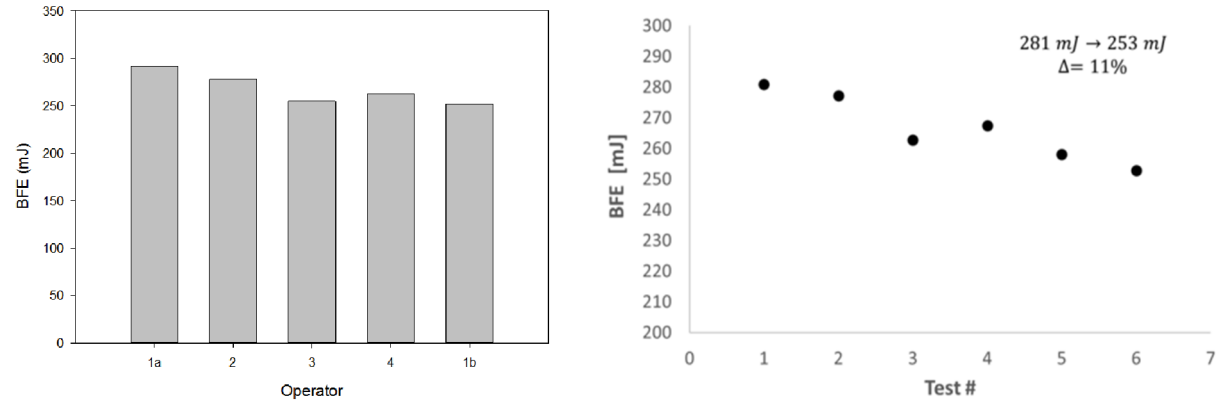

Figure 3: a) BFE measured by 4 different operators on the same sample of powder and b) effect of repeated measurements on the BFE with the same operator and sample of powder (transferred in a plastic dish between each test).

To understand the variation of the results, tests were repeated while leaving the powder in the rheometer vessel between each test. Figure 4 shows the variations of the BFE values during the repeats using

different types of powders. While the curves obtained during the stability tests (not shown here) were relatively flat and stable, an increase of the stresses was observed when repeating the complete test cycle. The relative augmentation of the stresses appears to be affected by the nature of the powder. Electrostatic charges measured on the SS 316 changed from +20 Volts before the test to + 2.7 Volts after the complete cycle. These measurements are an indication that tribocharging occurred during the tests and this affected the charge distribution in the powder. It is interesting to note that the variations observed when leaving the powder in the vessel are opposite to the ones witnessed when the powder is transferred from the vessel to a dish (plastic or metallic) between each repeat. While it is currently difficult to correlate the powder rheology with charge distribution, it is recognised that charges of particles may affect powder flow [14,15]. It is worth mentioning that no apparent modification of the apparent density or powder segregation was observed during the tests.

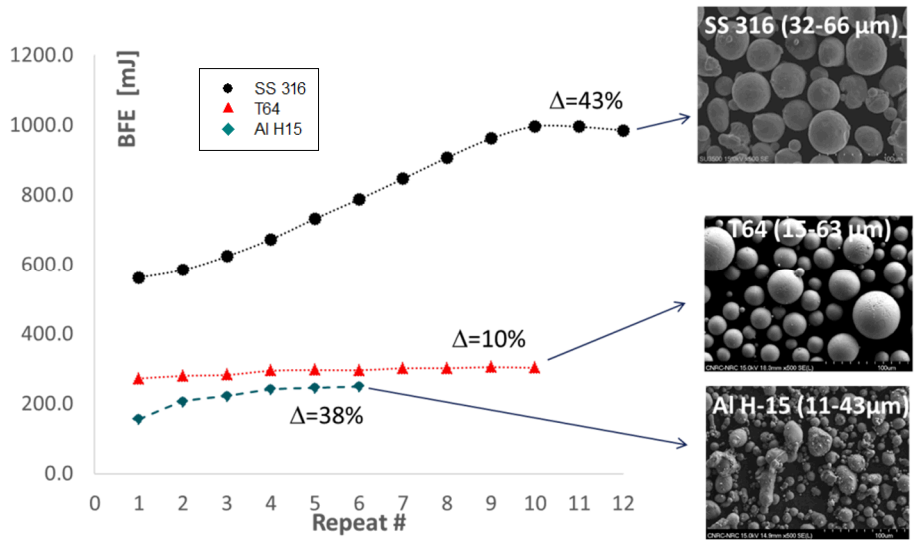

Figure 4: Effect of repeated measurements of the BFE on SS 316L (32-66µm), Ti6Al4V (15-63µm) and pure aluminium (11-43 µm) powders. Between each test, samples were left in the measurement vessel and reconditioning was done.

Effect of particle size distribution and composition

Figure 5a presents the effect of particle size on the basic flow energy of spherical pure aluminium powders with different median sizes (i.e. D_{50}) and narrow size distribution. The results show that the particle size has limited impact on the BFE. This appears to be counterintuitive as it is generally recognized that particle size significantly affects the flowability of powders and coarse powders tend to flow better than fine powders [2]. On the other hand, Figure 5b shows that the size distribution does have an impact of the BFE and the flowability energy seems to be larger with the powders with broader size distributions. One of the hypotheses could be the effect of the particle size distribution on the packing factor. However, apparent densities measured on the powders were very similar (1.54-1.55 g/cm^3) and do not support this hypothesis. Consequently, it would be valuable to conduct additional tests with other powders and size distributions to evaluate the sensitivity to particle size and materials.

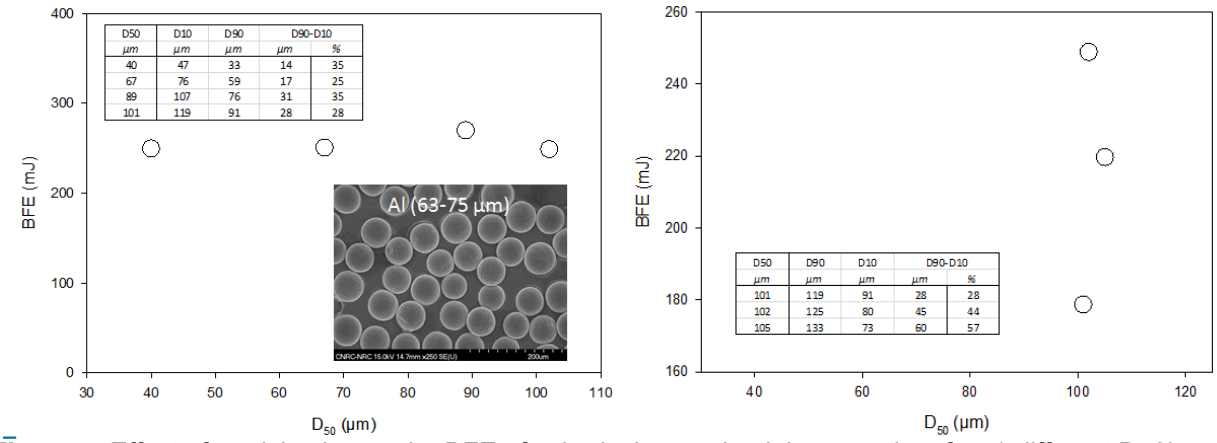

Figure 5: Effect of particle size on the BFE of spherical pure aluminium powders for a) different D50's with narrow particle size distribution and b) similar D50's with varying particle size distribution.

Figure 6 compares the Hall flow with the BFE index for CpAl and A357 aluminum powders of different particle size distributions. Figure 6a shows that the Hall flow tends to increase with particle size, which is contrary to the expected trend [2] and different from the BFE data observed with CpAl (Figure 5 and 6b). Figure 6 also shows that, while the effect of the alloy composition (CpAl and A357) is very small for the Hall flow (Figure 6a), significant differences are observed on the BFE (Figure 6b). This indicates that the two methods are not sensitive to the same particle features and provide different information on the flow behaviour. Figure 7 presents micrographs of CpAl and A357 powder having the exact same particle size distribution but different BFE indexes (data points encircled in Figure 6b). While both powders appear identical at low magnification on the SEM micrographs (high sphericity, few satellites and narrow particle size distribution), the images at high magnification show differences in surface topology. The cross sections (Figure 7 e and f) show that the shapes of the particles are also different and the A357 powder appears to be more spherical than the CpAl powder. The results suggest that the differences of surface topology and particle shape have a minimal impact on the Hall flow but the impact is significant on the BFE.

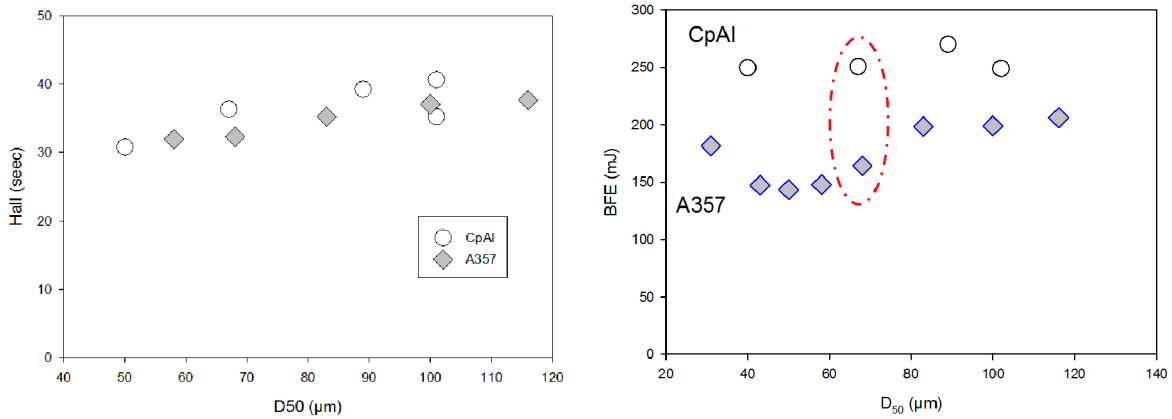

Figure 6: Effect of composition (CpAl and A357) and particle size distribution on a) Hall flow and b) BFE of spherical aluminium powders with narrow distributions (micrographs of the powders encircled in Figure 6b are provided in Figure 7).

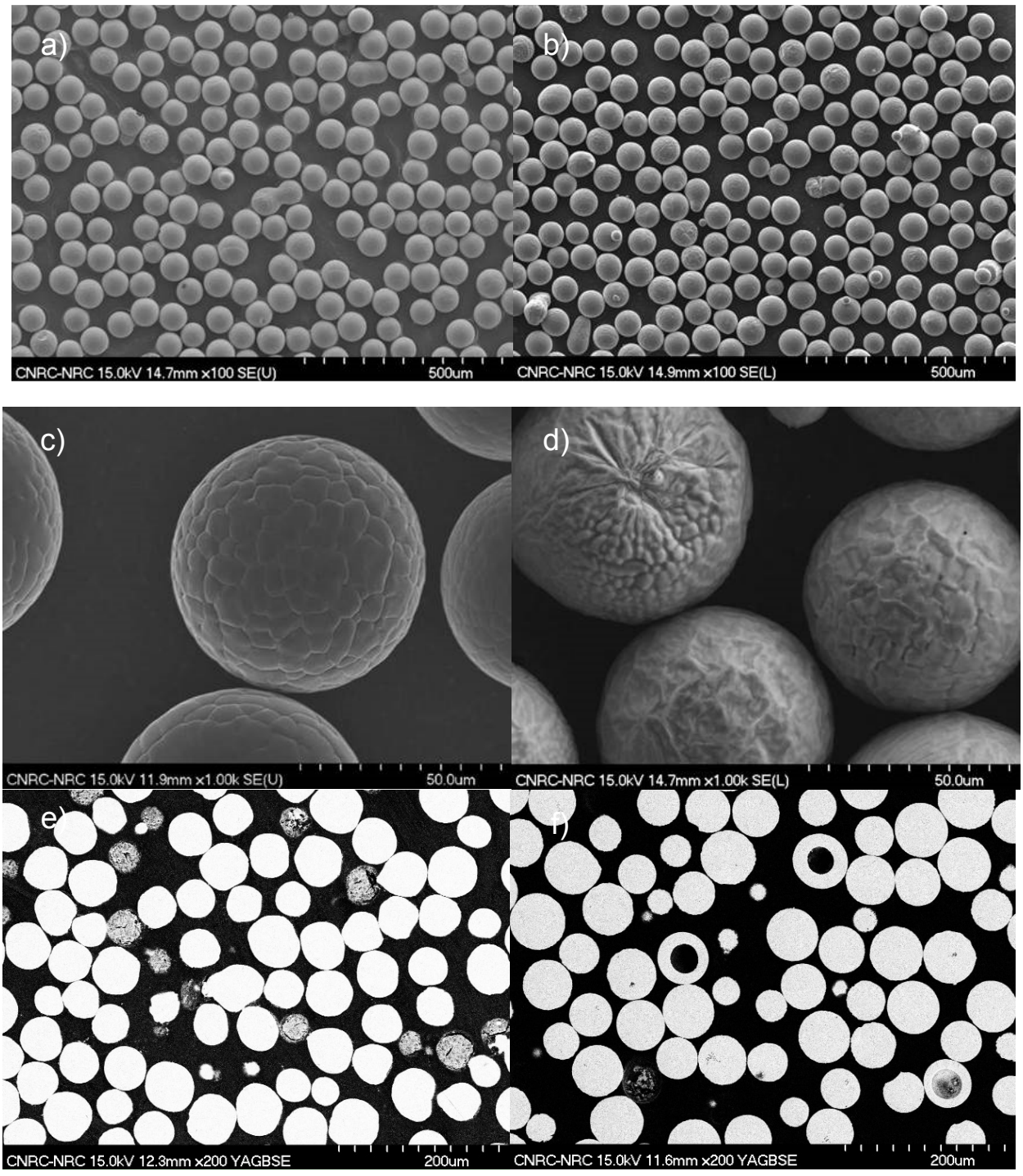

Figure 7: Microstructure of CpAl (left) and A357 (right) powders at low magnification (a, b), high magnification (c, d) and polished cross sections (e, f).

Effect of sphericity

Analyses were conducted to evaluate the variations of the sphericity of the different powders and assess its impact on the flowability measurements. Figure 8a shows that the sphericity of the A357 powders varies with particle size from 0.84 (90-106 μm) to 0.88 (53-63 μm) and that the BFE decreases as the sphericity increases, as anticipated. This can be associated to higher BFE values and Hall flow of A357 powder with larger particle size (Figure 6). The correlation between BFE and r_s is less clear but follows the same trend as the correlation between BFE and the sphericity. Figure 8b shows that the sphericity of the CpAl and A357 with the same size distribution are similar, but that the r_s of the A357 is higher than that of the CpAl, as suggested by the 2D cross sections showing more irregular particles for CpAl (Figure e and f). This is coherent with the BFE measurements and may explain the higher BFE measured on the CpAl powders. It should be noted that the large deviations are essentially due to the variations of the sphericity with the particle size distribution but also because not all sections analysed were taken at the equator of the particles. The effect of surface deformation on the measurement of sphericity will appear more significant when the section is closer to the surface of the particles.

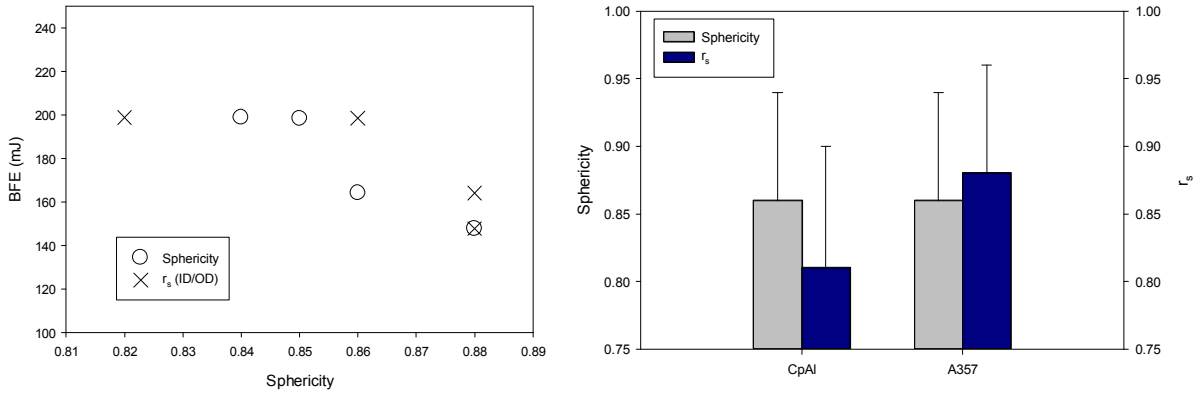

Figure 8: a) Effect of sphericity and r_s on the BFE of A357 powders with different median particle size and b) sphericity and r_s of CpAl and A357 powders with same size distribution (i.e. 63-75 μm).

Comparison of different flow indexes

Figure 9 presents the SE and NAS results obtained with CpAl and A357 powders having different median particle sizes. SE seems to have a low sensitivity to the median particle size when the particles are above a critical size of around 45 μm. However, when the particles are finer, higher SE values are obtained for both CpAl and A357. The effect of composition (CpAl vs A357) seems to be minimal for SE and NAS. Indeed, CpAl and A357 curves are superimposed, as observed with the Hall flow measurements (Figure 6a). For both SE and NAS, there are low consolidation forces during the tests and the measurements are not significantly affected by surface topology or particle shape for the powder investigated in the present study. The NAS, however, is strongly correlated with the size of the particles. The sensitivity of the NAS to particle size should come from the effect of the size of the particles on their fluidisation. Larger particles require higher air flow rates to get fluidized. It is, however, difficult to isolate the effect of gravity and surface interaction when comparing the results of powders with different particle size distributions. As the effect of particle size on SE was minimal (at least for particles larger than 45 μm), it is believed that the effect of the particle size on the NAS is essentially due to the effect of gravity.

Figures 10 and 11 present the correlations between different flow indexes. Figure 10 shows that there is no clear correlation between SE and BFE or NAS and BFE. While the forces were measured in all cases when the impeller was moving through a powder bed, the state of the powder and flow stresses differs significantly between the different tests and the response of the powder is very dependent on the flow conditions. The correlation between SE and NAS seems to be a little clearer, where SE appears to increase

with NAS. This is probably due to the fact that in both cases the powder is being tested in a nonconfined flow and there is no consolidating (as opposed to BFE).

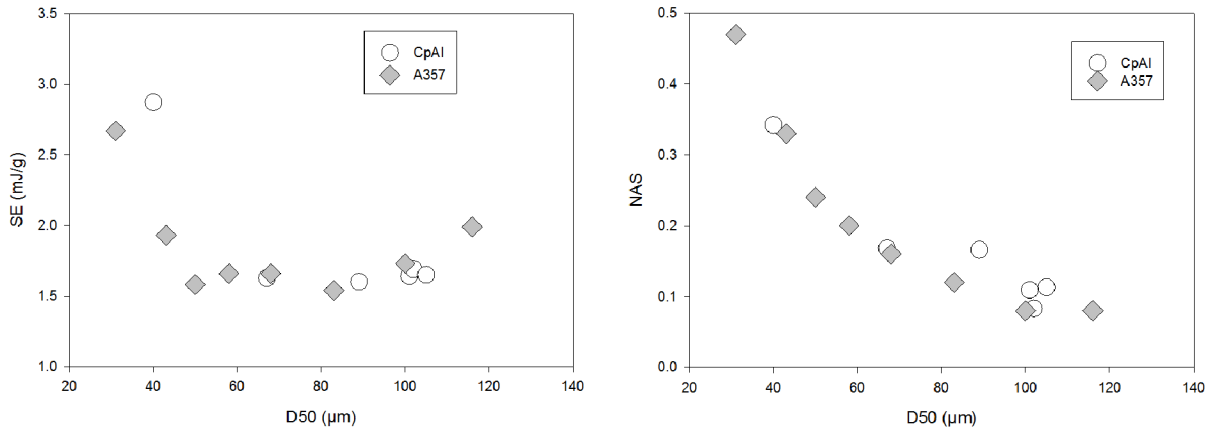

Figure 9: Effect of alloy composition (CpAl and A357) and particle size on a) SE and b) NAS measured on spherical aluminium powders with narrow size distributions.

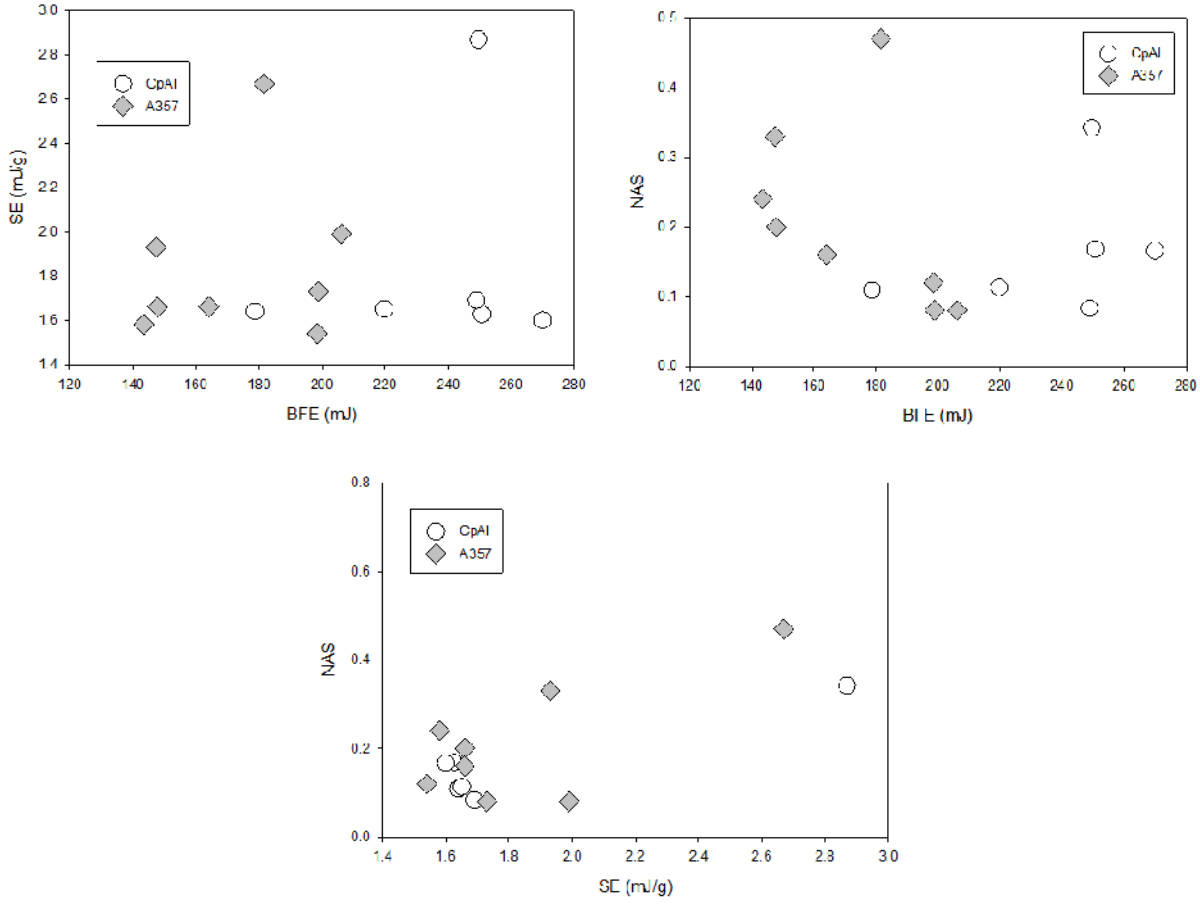

Figure 10: Correlation between a) SE and BFE, b) NAS and BFE and c) NAS and SE measured on CpAl and A357 spherical aluminium powders with narrow size distributions.

Figures 11a and 11b suggest that there is a correlation between the BFE and both the Hall and Carney flow. It is surprising to see that the data for CpAl and A357 are overlapped, since the BFE appeared to be

sensitive to the alloy composition (or surface topology/sphericity) as shown in Figure 6b while the Hall flow appeared to be unaffected by the composition. Figures 11c and 11d show that there is no apparent correlation between SE and both the Hall or Carney flow and no clear distinction between the CpAl and A357 results were observed.

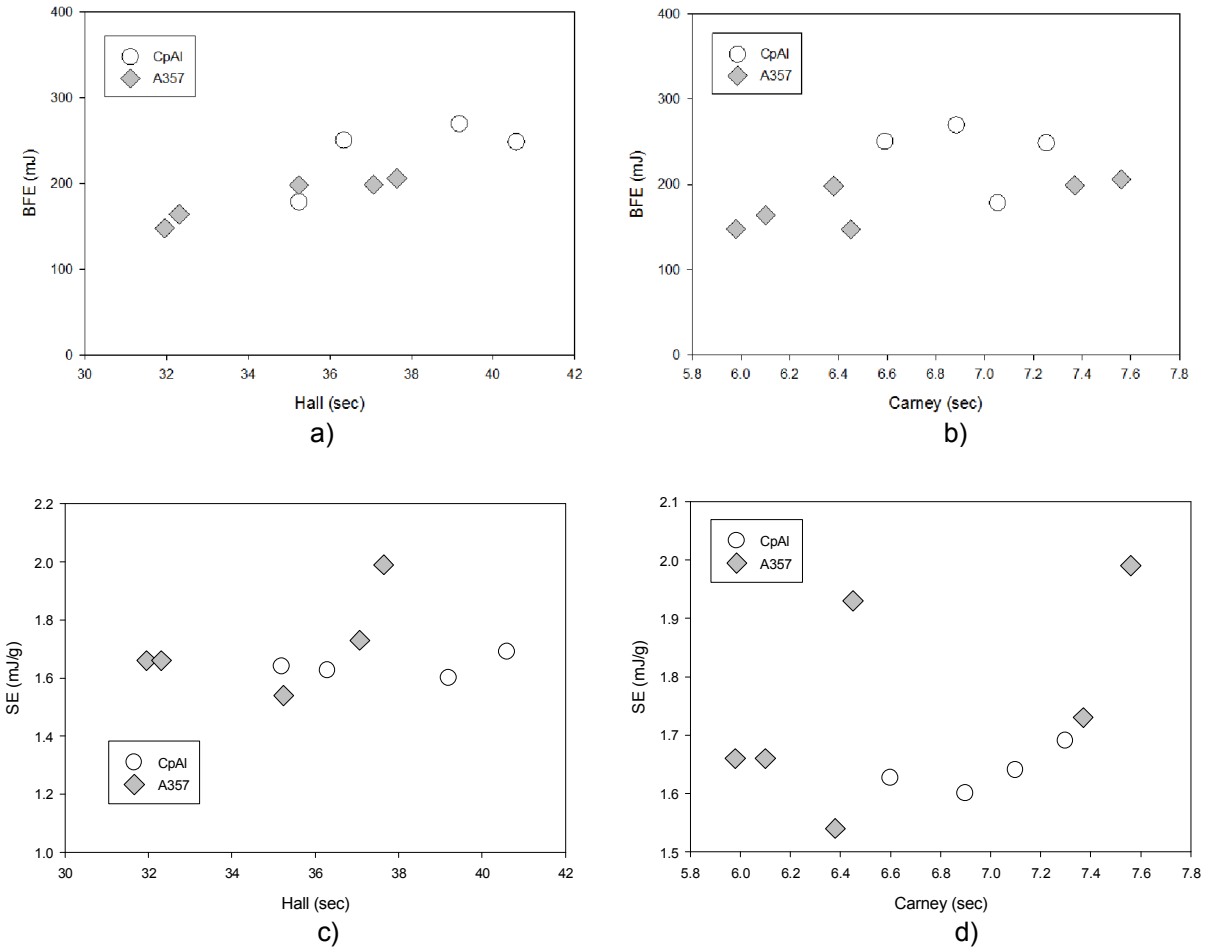

Figure 11: Correlations between a) BFE and Hall flow rates, b) BFE and Carney flow rates, c) SE and Hall flow rates and d) SE and Carney flow rates measured with CpAl and A357 powders with narrow size distributions.

Conclusions

This study shows that flowability of powders is affected by environmental, handling and measurement conditions, but correlations between different flow indexes are not always clear. As the tests were conducted under different flow conditions, the impact of measurement conditions and particle characteristics differs from test to test. The results showed that measurements obtained with a powder rheometer are affected by the friction forces between the particles but also by the friction between the particles and the vessel of the rheometer. This needs to be considered during the analysis of the results.

Flow measurement can be affected by the humidity present in the laboratory. The sensitivity to humidity depends on the type of test. Under low flow stresses (i.e. SE), humidity had an impact on the results evaluated with the rheometer. However, no effect was observed when the powder was in confined flow

under high compressive stresses (i.e. BFE). The adsorption of humidity happens rapidly and can be observed even if the powder is characterized directly after being collected from the bottle.

Measurements indicated that the flow behaviors of powders are modified during handling and testing. The variations observed seem to be associated with tribocharging during the tests. The results showed that not all powders have the same sensitivity to charging and rheology and flowability tests could be affected by the sensitivity to charging.

The effect of particle size distribution on the rheology of spherical aluminum powders (CpAl and A357) was not as anticipated. The median particle size (i.e. D50) did not significantly affect the flow energy of spherical CpAl particle with sizes ranging between 40 and 105 µm. Size distribution, surface roughness and sphericity may all have an impact on the flowability. Additional work is still required to understand how powder characteristics and measurement conditions affect flowability and establish how the different flow indexes are correlated with the powder processability and final part properties.

Acnowledgement

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