NRC Publications Archive
Archives des publications du CNRC

Assessing the robustness of powder rheology and permeability measurements

Lefebvre, L. P.; Whiting, J.; Nijikovsky, B.; Brika, S. E.; Fayazfar, H.; Lyckfeldt, O.

This publication could be one of several versions: author's original, accepted manuscript or the publisher's version. / La version de cette publication peut être l'une des suivantes : la version prépublication de l'auteur, la version acceptée du manuscrit ou la version de l'éditeur.
For the publisher's version, please access the DOI link below./ Pour consulter la version de l'éditeur, utilisez le lien DOI ci-dessous.

Publisher's version / Version de l'éditeur:
https://doi.org/10.1016/j.addma.2020.101203
Additive Manufacturing, 35, 2020-05-11

NRC Publications Archive Record / Notice des Archives des publications du CNRC :
https://nrc-publications.canada.ca/eng/view/object/?id=8394f3dc-c8c5-4f51-a6f6-cdaa6d6bc35f
https://publications-cnrc.canada.ca/fra/voir/objet/?id=8394f3dc-c8c5-4f51-a6f6-cdaa6d6bc35f

Access and use of this website and the material on it are subject to the Terms and Conditions set forth at
https://nrc-publications.canada.ca/eng/copyright
READ THESE TERMS AND CONDITIONS CAREFULLY BEFORE USING THIS WEBSITE.

L'accès à ce site Web et l'utilisation de son contenu sont assujettis aux conditions présentées dans le site
https://publications-cnrc.canada.ca/fra/droits
LISEZ CES CONDITIONS ATTENTIVEMENT AVANT D'UTILISER CE SITE WEB.

Questions? Contact the NRC Publications Archive team at PublicationsArchive-ArchivesPublications@nrc-cnrc.gc.ca. If you wish to email the authors directly, please see the first page of the publication for their contact information.

Vous avez des questions? Nous pouvons vous aider. Pour communiquer directement avec un auteur, consultez la première page de la revue dans laquelle son article a été publié afin de trouver ses coordonnées. Si vous n'arrivez pas à les repérer, communiquez avec nous à PublicationsArchive-ArchivesPublications@nrc-cnrc.gc.ca.

National Research Council Canada
Conseil national de recherches Canada

Assessing the Robustness of Powder Rheology and Permeability Measurements.

L.P. Lefebvre[1], J. Whiting[2], B. Nijikovsky[3], S.E. Brika[3], H. Fayazfar[4], O. Lyckfeldt[5]

[1] National Research Council Canada, 75 de Mortagne, Boucherville (QC), H1Y 2V9, Canada.
[2] Engineering Laboratory, National Institute of Standards and Technology (NIST), Gaithersburg, MD 20899, USA
[3] Laboratoire sur les alliages à mémoire et les systèmes intelligents (LAMSI), École de technologie supérieure ,1100 Notre-Dame Ouest, Montréal (QC), Canada H3C1K3
[4] Multi-Scale Additive Manufacturing Lab, Mechanical and Mechatronics Engineering Department, University of Waterloo, Waterloo, ON, N2L 3G1, Canada
[5] RISE IVF AB, Argongatan 30, SE-431 53 Mölndal, Sweden
Corresponding author: Louis-Philippe Lefebvre: louis-philippe.lefebvre@cnrc-nrc.gc.ca, 450-641-5031,

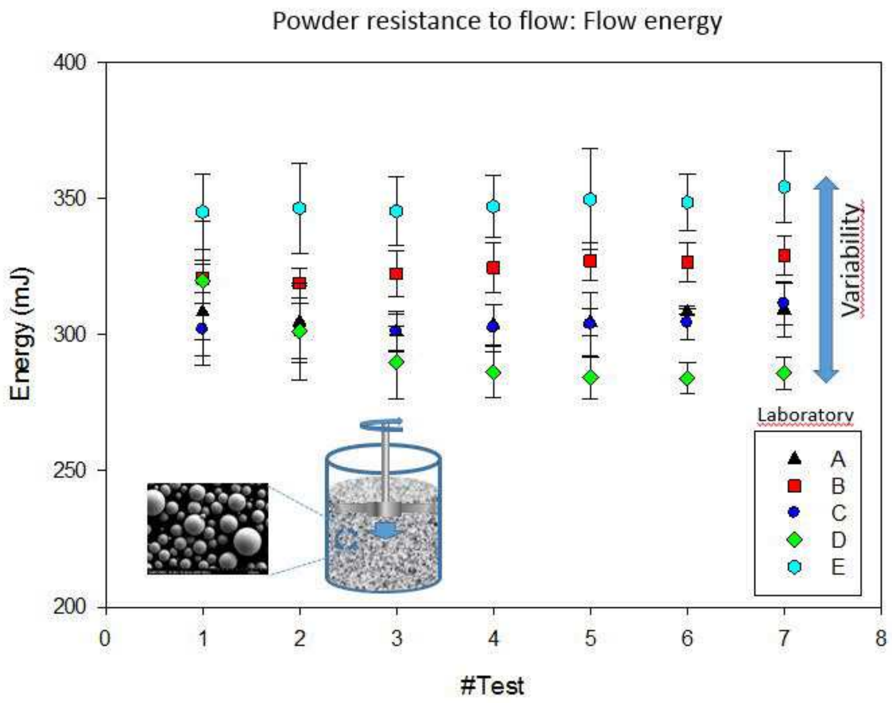
Highlights:

Factors affecting the powder rheology measurements and results variability.

Abstract

Powder metallurgy processes rely on powder flowability. However, flowability is not an intrinsic property and depends on the measurement conditions. Standards have been developed to adjust measurement methods to various flow conditions but there is presently questions whether current methods are adapted to the specific requirements of powder bed additive manufacturing. Rheology has been used to assess powder flowability but there is still limited information available on the robustness of the method. This paper presents the flow characteristics measured in five laboratories with a powder rheometer. Attempts were made to understand the sources of intra and inter laboratory variations and find ways to reduce them. The variations do not seem to be associated with sampling or environmental conditions. Experimental setup, calibration and/or the modification of the powder during handling could be associated with the variations observed. However, additional tests would be required to confirm the sources and improve the repeatability of the measurements.

Graphical Abstract



Keywords: Powder flowability, rheology, round robin, reproducibility, repeatability.

1. Introduction

There is an interest to quantify how powder flows and associate the values measured with powder behavior during a fabrication process [1]. Flowability is affected by many powder characteristics (e.g., particle size distribution, morphology, presence of satellites, density, and surface interaction) as well as the environmental and handling conditions (e.g., humidity, temperature, atmosphere, and measurement method). Depending on the manufacturing process, powders are exposed to different flow conditions, including plastic, inertial, fluidized, and entrained flow [2].

It is recognized that powder flowability has an important impact on additive manufacturing (AM) processes. For the powder bed fusion process, part quality relies on the characteristics of the powder layer (conductivity, density, uniformity) spread during the manufacturing process and the presence of defects in the powder layer. Besides, build rate and productivity are affected by the time to spread the powder layer, which is a function of the powder flowability. However, the correspondence between powder performance in powder bed fusion machines and powder properties is not yet understood and therefore cannot be used to control final part properties. AM machine users have reported that powders appearing to be identical when measured using standardized methods may behave differently in the machines. Recently, America Makes & ANSI AMSC Standardization Roadmap for Additive Manufacturing [3] reported that existing standards for flowability do not account for the range of conditions that a powder may encounter during the AM processes and R&D is needed to measure and quantify flowability, and increase process productivity and robustness. Therefore, reliable measurement methods are required for the wide acceptance and adoption of AM technologies by industry.

Quantification of the flowability of powders is important for different reasons including quality control, powder selection, certification, simulation, research and product development. For qualification and quality control purposes, the method must be simple, robust and widely available. In addition to these requirements, flexibility and precision are required for research activities. Different techniques have been developed to quantify powder flowability. The most common methods are based on funnel discharge time measurements such as the Hall flowmeter. The methods are covered by various standards (MPIF [4], ATSM [5] and ISO [6]) and are extensively used in industry. One of the advantages of these methods is their simplicity and accessibility (i.e., relatively low cost and rapid compared with more sophisticated methods). One of the limitations of the approach comes from its inability to quantify the flowability when the powder doesn't flow through the orifice of the funnel. Other funnel flow meters (Carney, Gustavsson) have been developed to compensate for this limitation. These techniques reproduce relatively well the way the powder flows in an open funnel. However, they do not correspond to the conditions encountered in various processes (e.g., spreading thin layers of powder as is the case in the powder bed fusion process). The Hausner Ratio (ASTM D7481 [7]), defined as the ratio of the tapped density and the apparent density, has also been used to quantify powder cohesion.

B.H.Kayes [8,9] studied the avalanching behavior of a powder using a rotating disc filled with powder and described the pattern of events generated by an avalanching powder using fractal geometry. A strange attractor plotted in discreet time maps were used to evaluate the effect of particle size distribution, humidity, and temperature on the rheological behaviour of powders. Avalanche concept was recently adapted to develop commercial systems [10,11] that have generated interest in the powder metallurgy community. Different flow indicators such as the avalanche angle or dynamic angle of repose, avalanche energy, surface fractal and linearity, or deviations in any of these metrics can be used to quantify the flow characteristics of the powders.

The flowability can also be evaluated by measuring the cohesion between particles using rheology approaches, by measuring the resistance of a powder to flow (e.g., resistance seen by a blade when moving through a cylinder filled with powder) or shear tests (ASTM D6467 [12] and D3080/D3080M [13]). Commercial equipment has been developed to conduct such tests [14,15,16]. These methods have recently generated interest to qualify the flowability for powder metallurgy applications and additive manufacturing.

In order to use flowability tests for quality control, certification, simulation, research and product development, it is essential to evaluate the repeatability and robustness of the methods. Data obtained in an interlaboratory study performed by seven laboratories and presented in the ASTM B213-17 standard indicated that the relative reproducibility of Hall flow can be as high as 21 % [5]. More recently, results obtained by 19 different laboratories in a proficiency program organized by the ASTM B09 committee showed a large span in the results and relative standard variations (average 32.4 s ± 5.9 s) with AM grade Ti6Al4V powders [17]. These variations may be problematic when comparing results from different laboratories or if results cannot be reproduced within a laboratory.

The objective of the work presented in this paper was to investigate the inter-laboratory variability of the rheology method to quantify the flowability of a Ti6Al4V powder (15 µm to 63 µm sieve range), typically used in powder bed fusion AM processes. Both resistance to flow and permeability were evaluated. The tests were conducted in different laboratories and were measured by different operators using the same equipment and procedures.

2. Experimental Procedures

All tests were conducted with a plasma atomized Ti6Al4V grade 23 (15 µm to 63 µm sieve range) powder (from AP&C, a GE additive company[1]). Scanning electron micrographs of the powder were taken at different magnifications using a Hitachi S-4700 scanning electron microscope. The particle size distribution was evaluated using laser diffraction (Beckman Coulter LS 13 320, 0.4 µm to 2000 µm range, dry measurement using the Tornado DPS module). The powder, from the same batch, was split into five polyethylene bottles (1.5 kg each) using a 2-sides splitter (i.e., SP-171X from Gilson) employing a combination of splitting and recombination to make sure the samples were similar and homogeneous. A bottle was sent to five different laboratories to conduct different tests (stability, variable flow tests, aeration and permeability) using the same model of powder rheometer (FT4 from Freeman Technology, a Micromeritics company). The five laboratories that participated in the study were the National Research Council Canada (Canada), the National Institute of Standards and Technology (USA), the Multi-Scale Additive Manufacturing (MSAM) at the University of Waterloo (Canada), RISE AB (Sweden), and the École de Technologie Supérieure (Canada).

All rheology tests were done in triplicate after conditioning the powder using the instrument manufacturer's suggested methodology. The rheology tests were conducted using a 25 mm x 25 ml split vessel. The splitting device, provided with the instrument, is intended to provide a fixed volume and mass of powder. The conditioning, recommended by the rheometer manufacturer, consisted of gently disturbing the powder using the blade as it enters in the powder and rotating clockwise to create a lightly packed test sample prior to testing. This conditioning was used to prepare the sample in a reproducible manner, removing stress history or excess air prior to the measurements and to minimise the effect of handling the powder prior to the measurements (i.e., reduce operator to operator variability).

Flow tests (stability, variable flow and aeration) were evaluated by measuring the powder resistance when moving a rotating stainless-steel blade (23.5 mm diameter) in a glass cylinder (borosilicate, 25 mm internal diameter) filled with powder (see Figure 1). The powder resistance was measured using torque (resolution 0.002 mNm) and linear force (resolution 0.0001 N) sensors. The energy (integral of the energy gradient over the distance travelled) was calculated

---

[1] Certain commercial entities, equipment, or materials may be identified in this document in order to describe an experimental procedure or concept adequately. Such identification is not intended to imply recommendation or endorsement by the participating laboratories, nor is it intended to imply that the entities, materials, or equipment are necessarily the best available for the purpose.

using the resultant of the torque and linear forces as the blade was moving downward and forcing the powder towards the bottom of the cylinder.

The stability tests were done by measuring the flow energy for seven identical repeat tests on each sample, with conditioning of the powder prior to subsequent tests. The speed of the tip of the blade was fixed at 100 mm/s. The Basic Flowability Energy (BFE) index, used as a measure of powder flowability, represents the energy measured at test repeat #7, as set in the FT4 procedure. The sensitivity of the flow energy to shear rate (see Figure 2) was evaluated using the variable flow rate tests (energy vs tip speed). These tests were conducted by measuring the flow energy with tip speed varying from 100 mm/s down to 10 mm/s. The flow rate index (FRI), defined as the ratio of the values measured at 10 mm/s divided by the value measured at 100 mm/s, was used as an index of the sensitivity of flowability to the shear rate (i.e., tip speed). Stability and variable flow rate tests were done in the same cycle but the results are presented separately in the results section.

The sensitivity of the flowability to air flow was evaluated using aeration tests. These tests were done by measuring the flow energy as a function of dry air flow rate introduced through a porous mesh at the base of the cylinder (35 ml). The flow energy was measured in a similar way to the stability and variable flow tests. The range of air velocity was varied from 0 mm/s to 10 mm/s, with a conditioning before each test cycle. The maximum of 10 mm/s was selected to minimise the formation of dust clouds out of the cylinder during the measurements. The aeration ratio was evaluated at two air flow rates (4 mm/s and 10 mm/s). AR_4 was derived by dividing the flow energy measured at 0 mm/s by the value measured at 4 mm/s while AR_10 was derived by dividing the flow energy measured at 0 mm/s by the value measured at 10 mm/s.

The resistance to air flow was measured by evaluating the pressure drop across the volume of powder while air was flowing (at a constant 2 mm/s) through the powder-filled cylinder (25 mm diameter, 10 ml glass vessel). The powder was subjected to varying vertical loads via a vented piston (i.e., applied consolidation pressure). The measurements were done under different applied consolidation pressures (0 kPa to 15 kPa). The results are reported as the pressure drop as a function of the applied consolidation pressure. Values measured at 15 kPa were compared to assess the reproducibility of the measurements.

For all tests, three specimens (i.e., three different powder samples) were characterised in each laboratory using the manufacturer’s recommended procedure. A first set of experiments, henceforth referenced as round 1, were conducted with no other recommendations than those provided in the FT4 user manual. A second series of tests, henceforth referenced as round 2, was subsequently conducted using a procedure with tighter constraints for the operator to see if the variability could be reduced. Also, during these tests, each laboratory recorded the temperature and humidity level with a hygrometer. Standard deviations reported were calculated using the Bessel's correction to provide an adjustment for the bias associated with the small sample data (3 data point per conditions). Deviations were also calculated for all BFE data points (30 points: 3 replicates in 5 laboratories measured in 2 rounds).

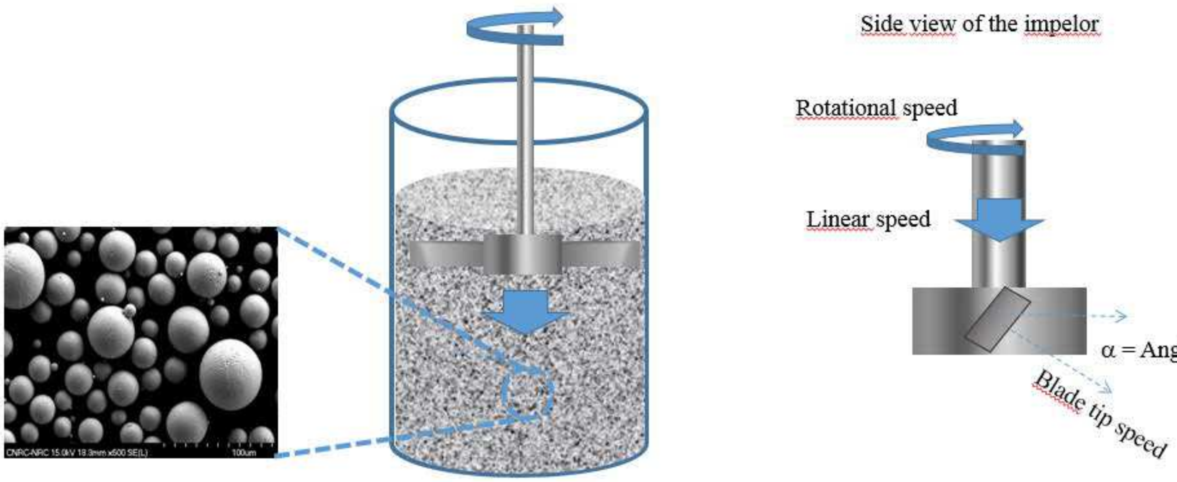


Figure 1: Schematic description of the method to measure the flow energy showing the movement of the blade and the method to calculate the tip speed. [18]

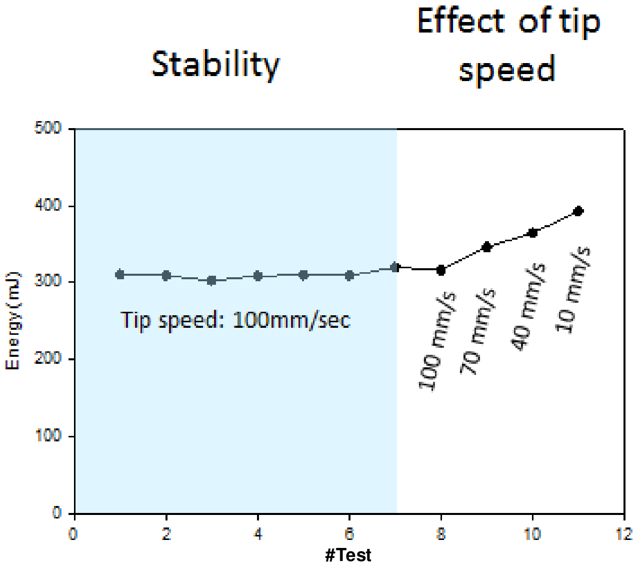
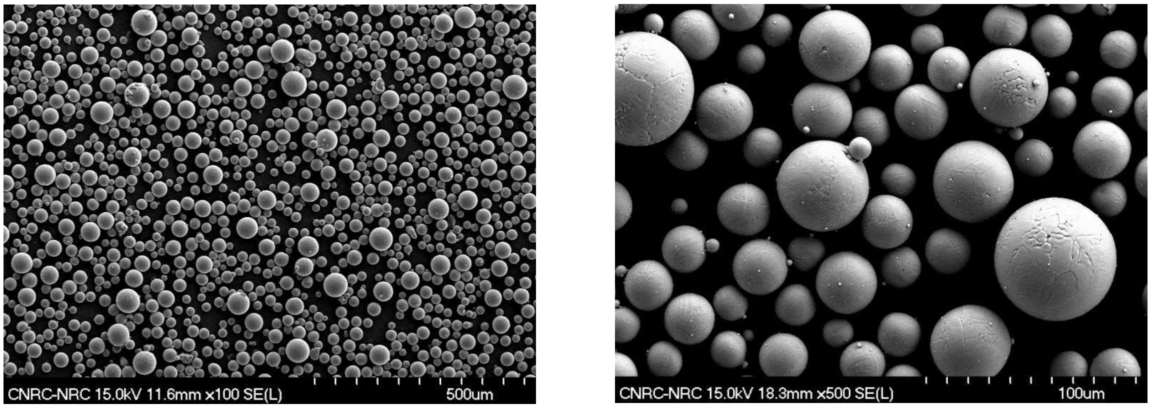


Figure 2: Schematic representation of the results obtained with the FT4 rheometer. The first seven measurements are conducted at constant tip speed of 100 mm/s to evaluate the stability of the measurements. The seventh measurement was used as the basic flow energy (BFE) index. Measurements 8 to 11 were obtained by varying the tip speed from 100 mm/s down to 10 mm/s to investigate the effect of shear rate. While the stability and variable flow tests were conducted during the same test, they are reported separately in the result section.

3. Results.

Figure 3 presents low (100x) and high (500x) magnification images of the powder. Images show that the powder is highly spherical with minimal satellites. The laser diffraction particle size distribution of the powder is shown in Figure 4. The measured particle size distribution agrees roughly with the sieve range provided by the manufacturer (15 µm to 63 µm). Table 1 presents different indices measured in the 5 laboratories in round 1.



a) b)

Figure 3: SEM micrographs of the Ti6Al4V powder used in this study: a) low magnification and b) high magnification.

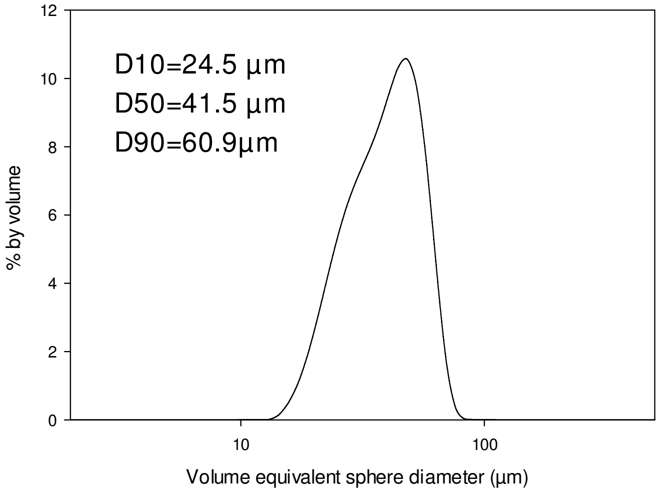


Figure 4: Particle size distribution of the powder investigated in this study (measured by Laser Diffraction, Beckman Coulter LS 13 320) with corresponding D10, D50 and D90 that represent the intercepts at 10%, 50% and 90% of the cumulative mass (dry measurement).

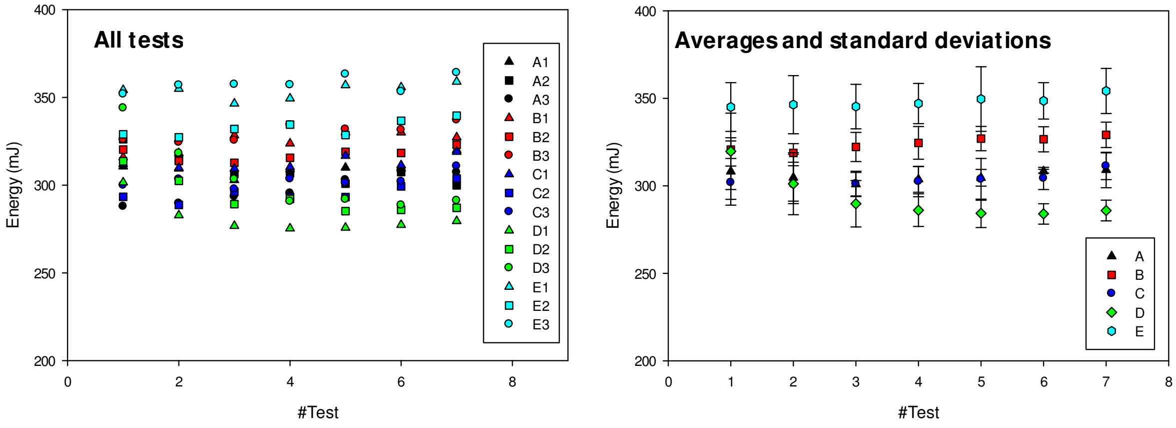
Figure 5 presents the results of the stability tests measured in the different laboratories (Figure 5a presents all results and Figure 5b the averages and standard deviations in each lab). The results indicate that the values are relatively stable during the test (curves are flat and the energy does not vary a lot during a test). Indeed, Figure 6a shows the stability indices (i.e., value measured during test #7 divided by value measured during test #1) are close to one for most laboratory.

PDF-XChange Product Click to buy NOW! www.tracker-software.com

This suggests that the powder is relatively stable and not significantly altered during the tests by consolidation, attrition, agglomeration, electrostatic charging, shear and/or humidity sorption/desorption. However, there are differences between the laboratories: the inter-laboratory variability is higher than the intra-laboratory variability. This observation is coherent with the comparison of the basic flow energy index (BFE, test #7 reported in Figure 6b for all laboratories) that shows there are variations from lab to lab and these variations are more important than those measured within each laboratory. When analysing all BFE results (three measurements done in the five different laboratories), the values range from 279 mJ to 364 mJ, with an average of 318 mJ ± 25 mJ (see Table 1). The spread between the minimum and maximum values (85 mJ) is significant and represent a relative variation ((Max-Min)/Average) of 27 %.

Table 1: Basic flow energy (BFE), the flow rate index (FRI), the aeration index (AR_4 representing the value measured at v = 0 mm/sec divided by the value measured at 4 mm/sec) and pressure drop measured at 15 kPa in the different laboratories in round 1.

Laboratory	BFE (mJ)		FRI		AR_4		Pressure drop @ 15 kPa	
	Av	Stdev	Av	Stdev	Av	Stdev	Av	Stdev
A	309.1	10.2	1.21	0.05	54.6	6.0	6.27	0.32
B	329.2	7.3	1.21	0.01	69.3	17.6	6.34	0.89
C	311.0	7.6	1.20	0.01	56.1	10.7	6.44	0.06
D	285.9	5.9	1.24	0.02	44.2	11.6	6.10	0.04
E	354.2	12.9	1.19	0.01	56.3	11.8	6.28	0.07
Average	***317.9***		***1.21***		***56.1***		***6.29***	
Stdev	***25.5***		***0.02***		***10.3***		***0.12***	



a) b)

Figure 5: Stability test results (round 1) a) all data points recorded by the different laboratories and b) averages and standard deviations for each laboratory.

PDF-XChange Product
Click to buy NOW!
www.tracker-software.com

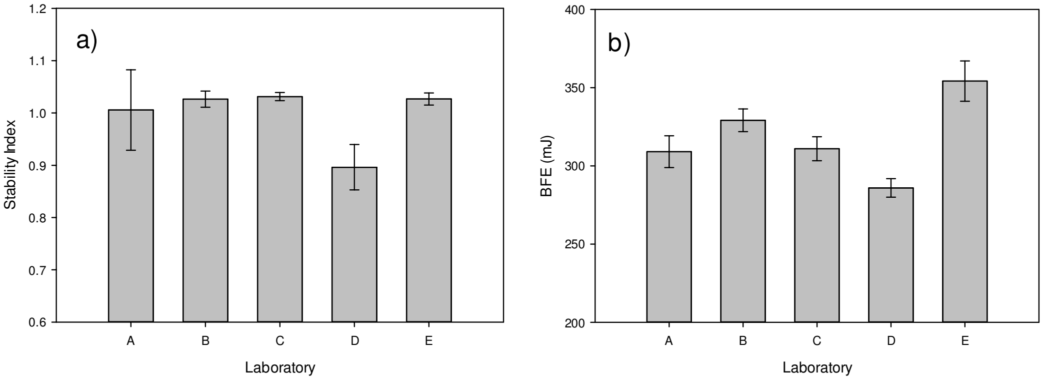
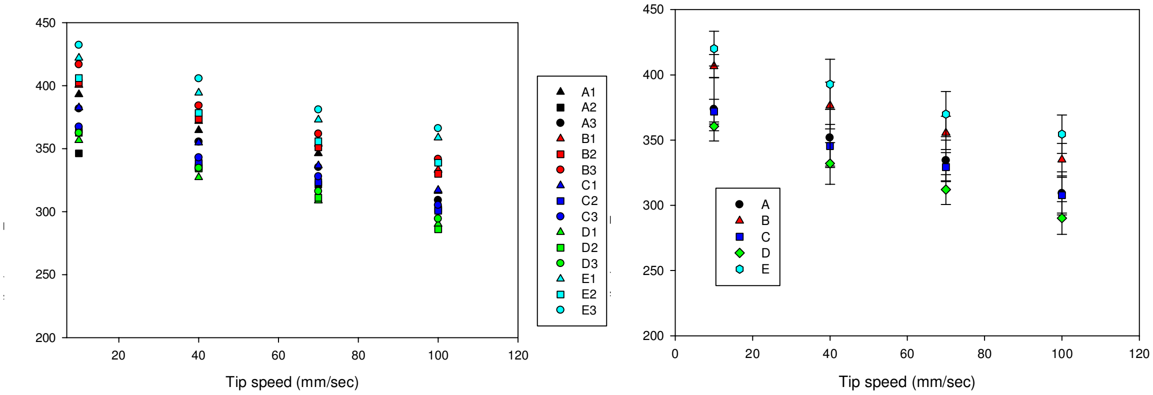


Figure 6: a) Stability index (energy at Test #7 divided by the energy at Test #1) and b) averages and standard deviations of BFE index (Test #7 in figure 5b) measured by all laboratories in round 1.

The effect of the tip speed is presented in Figure 7. While the effect of the shear rate appears to be similar for all labs (i.e., all measurements show the same trend), a spreading of the results is observed, for reasons similar to those observed in the stability tests (Figure 5). Indeed, the variations at 100 mm/s are similar to those observed in the stability test. Once again, the inter-laboratory variations are greater than the intra-laboratory variations. The Flow Rate Index (FRI, Figure 8) is similar for all labs with low standard deviations, confirming that the effect of the tip speed was similar for all tests.



a) b)

Figure 7: Effect of the tip speed on the flow energy a) all data measured and b) averages and standard deviations measured by the different laboratories.

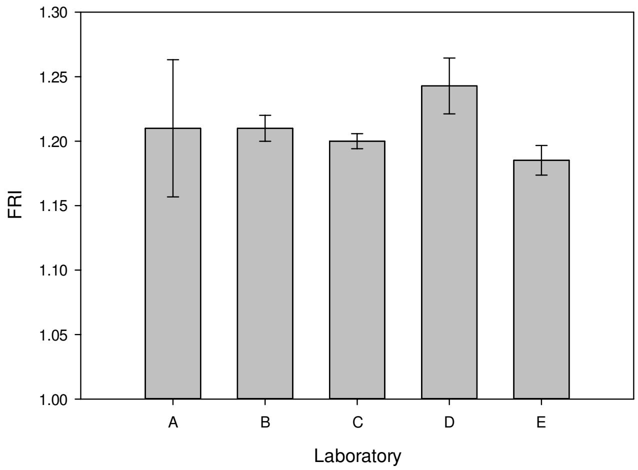


Figure 8: Flow rate index (averages and standard deviations) obtained in the different laboratories. The flow rate index represents the flow energy measured at 10 mm/s divided by the energy measured at 100 mm/s.

The effect of air flow on the energy required to move the blade through the powder is presented in Figure 9. The air flow range was selected to achieve significant variations in energy while avoiding particles to flow out of the cylinders. The conditions allow a decrease in energy to be observed followed by a plateau, where the impact of further increases in air flow on energy is minimal. The flow energy is significantly reduced when air is injected at the bottom of the cylinder, as expected. This is caused by the fluidisation of the powder, which results in an increase of the distance and a reduction of the forces between the particles. The conditions allow observing a rapid decrease followed by a plateau, where the impact of the increase of air flow on the energy is minimal.

The results presented in Figure 9 show significant variations. At 0 mm/s, the energy varies from 301 mJ to 627 mJ (average 417 mJ ± 106 mJ). This variation is much greater than that observed during the stability tests (Figure 5). Indeed, the variation (Max-Min = 326 mJ; representing a relative variation of 78 %) is significantly greater than the one observed with the BFE (i.e., 27%). In addition, the average value (417 mJ) is significantly different from the average BFE (318 mJ, Figure 6b). It was observed that not all laboratories used the same procedure. The rheometer manufacturer recommend to avoid splitting the powder before these tests and use the same amount of powder after splitting measured during the stability tests. However, some laboratories used the amount of powder before splitting while others used the amount after splitting. Consequently, not all tests were conducted with the same amount of powders (i.e. the powder mass was varying between 67.3 to 79.8 g). Analysis of the results showed there was a correlation between the force measured and the mass of powder and the variation of the mass could be a source of the difference observed during these tests (see discussion section).

When the powder is fluidised, the forces gets small and variation appears more significant. For the powder and conditions investigated, this causes large relative variations in the AR_10 results (67 to 159) which represents the energy measured at 0 mm/s divided by the energy measured at 10 mm/s. When measuring the aeration ratio at 4 mm/sec (in the linear portion of

PDF-XChange Product Click to buy NOW! www.tracker-software.com

the energy vs air velocity curve), variations are significantly smaller and aeration ratios ranging from 44 to 69 are calculated. Obviously, the absolute AR_4 and AR_10 values are different as these values represent ratios of energies measured under different conditions. Therefore, these indices should only be used to compare powders if measurements are made under similar conditions.

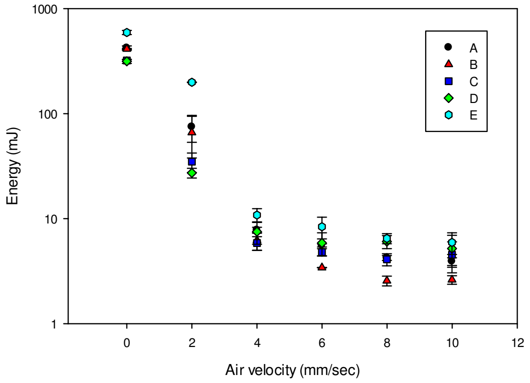
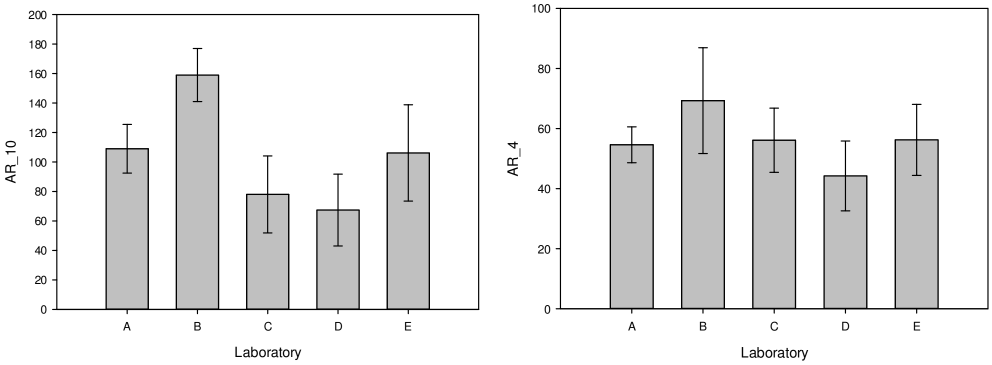


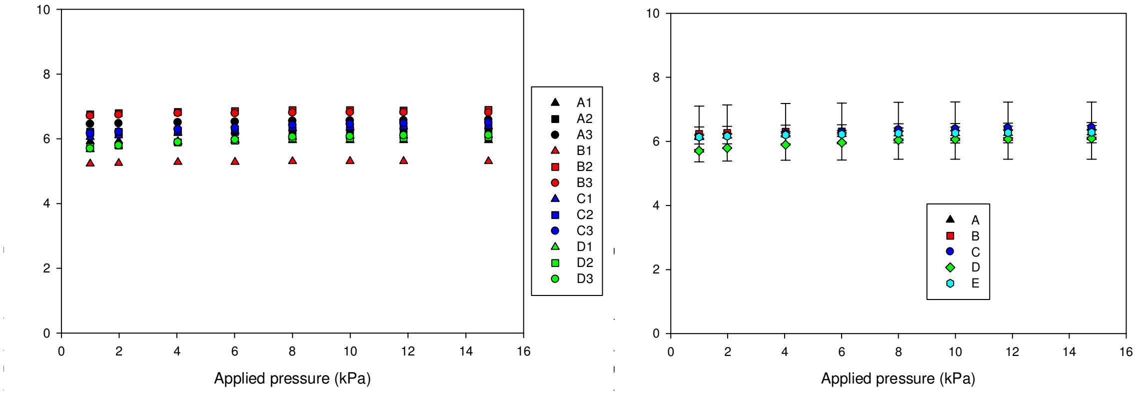
Figure 9: Effect of air velocity on the flow energy (averages and standard deviations) presented on a log scale.



a) b)

Figure 10: Aeration ratio (averages and standard deviations) obtained in different laboratories a) energy measured at 0 mm/s divided by the energy measured at 10 mm/s (AR_10) and b) energy measured at 0 mm/s divided by the energy measured at 4 mm/s (AR_4).

Permeability results presented in Figure 11 and 12 show that the air pressure drop through the powder is not significantly affected by the applied pressure. Indeed, the consolidation of the powder is minimal under the pressures used in the tests and once the powder is packed (already observed under 1 kPa), the density of the powder and permeability are little affected by the pressure. Variations were observed in the different laboratories (1.58 mBar spreading observed at 15 kPa). Standard variations of all results equals 6 % while the standard variations of the average of all laboratories was 0.12 mBar, representing 2 % of the average of all data at 15 kPa.



a) b)

Figure 11: Pressure drop as a function of applied pressure, a) all data measured and b) averages and standard deviations measured by the different laboratories.

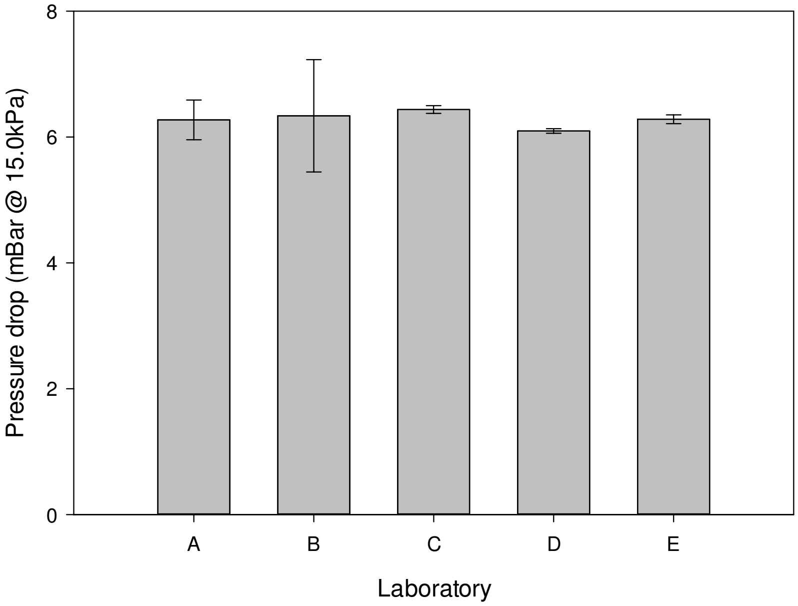


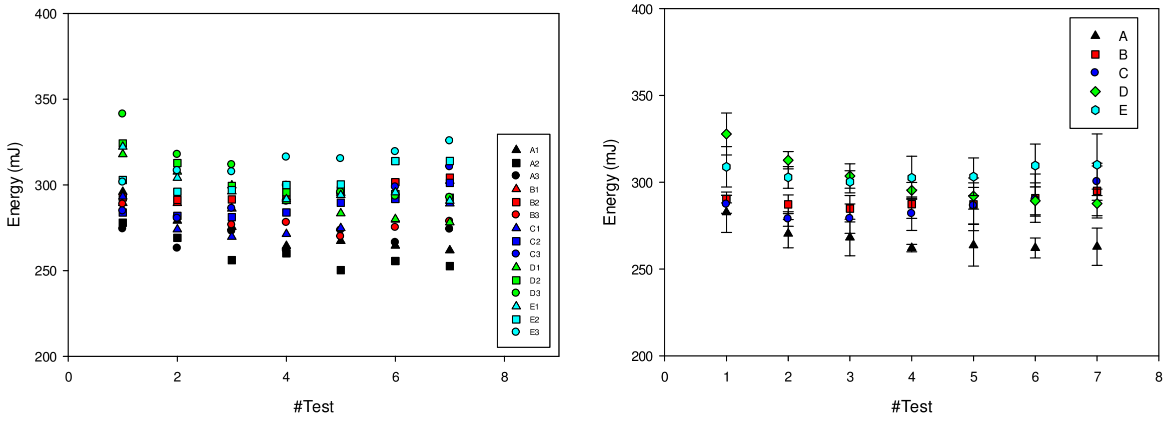
Figure 12: Pressure drop (averages and standard deviations) measured at 15 kPa by the different laboratories.

To minimize the variations coming from the handling of the powder, a test procedure was prepared (Table 2) and a new series of stability tests was conducted in the different laboratories (round 2). The results obtained (Figure 13) show that the new procedure did not allow reducing the variations between the different laboratories. In fact, a BFE average of 291 mJ ± 20 mJ was obtained with results spanning from 253 mJ up to 326 mJ (representing a relative variation of 25 %). Interestingly, the BFE values obtained in the second round are lower than those obtained in the first series of tests. Besides, there is an apparent correlation between the values measured during the two rounds (Figure 14b).

PDF-XChange Product Click to buy NOW! www.tracker-software.com

Table 2: Procedure developped by the different participating laboratories to minimise the measurement variations for the stability test.

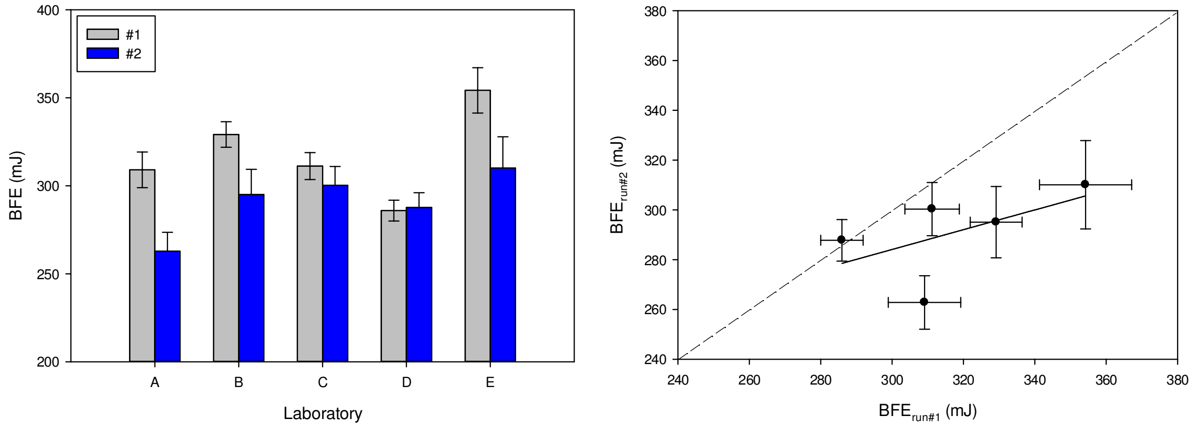
Equipment: Freeman FT4
Calibration: To be done before initiating the tests
Operator: All tests should be done by the same operator in each laboratory
Handling/sampling
- Mix the powder in the as received HDPE bottle with a metallic spoon (30 sec by hand) prior to transfer to the glass cylinder (see next section).
<u>Note</u>: The powder should be kept in small metallic cans (tin coated steel can) after the test in case additional analyses are required.
Testing (stability test):
- Clean the instrument before each test (see next section).
- Install the blade.
- Place the fully assembled empty vessel, without the funnel on to the FT4 Table. When the balance has settled, click the 'Tare Empty Vessel' button.
- Place the funnel and fill the vessel with the sample: After mixing the powder in the as received HDPE bottles with a metallic spoon (see previous section), transfer 75 ± 0.1g the powder using the metallic spoon into the glass cylinder (25 ml, 25 mm diameter glass cylinder, new powder used for every test). Fill the cylinder with care to avoid uneven filling of the cylinder.
- Remove the funnel and return the filled vessel to the FT4 Table.
- Screw the clamp to secure the vessel to the FT4 Table.
- Put the protection screen.
- Start the test (condition the powder using FT4 procedure).
- Split the vessel. Use a plastic tray to collect the excess powder from the splitting process. Collect the recovered powder and keep the powder in a metallic can (tin coated steel can[1]).
- Remove excess powder on the splitter using a brush (use separate brush if testing different powders), and return the top of the splitter to its original position.
- Continue the test immediately after using standard test program supplied with the FT4 powder rheometer.
- Note temperature and moisture prior to each test.
- Conduct all tests with the same set–up (cylinder, clamping, splitter…).
- Save the results.
- Repeat the entire procedure (with cleaning between each test) 3 times to assess reproducibility. Mix the powder prior to each test as described in the Handling/sampling procedure.
Cleaning procedure
- Between each test, clean the entire vessel (including glass cylinder, Delrin base and hinge mechanism) to remove traces of powder left on the surface of the cylinder using the following procedure:
- Dry wipe with Kimwipe (Kimtech Science Brand, Kimberly-Clark Professional) to remove the excess of powder.
- Clean again with Kimwipe and isopropanol.
- Dry wipe again with dry Kimwipe to remove excess isopropanol and dry the cylinder (air dry for 10 min).
- Between each test, remove the blade, clean it with a brush, rinse it with isopropanol and gently dried it with Kimwipe.
<u>Note</u>: Make sure all pieces of the instrument are dry and free of isopropanol prior to conduct the test (air dry 10 min).



a) b)

Figure 13: Stability test results (round 2) a) all data points recorded by the different laboratories and b) averages and standard deviations for each laboratory.

PDF-XChange Product
Click to buy NOW!
www.tracker-software.com



a) b)

Figure 14: Comparison of the basic flow energy (BFE) data measured during round 1 and 2 a) averages and standard deviations and b) correlation between the results obtained in round 1 and 2 in the different laboratories.

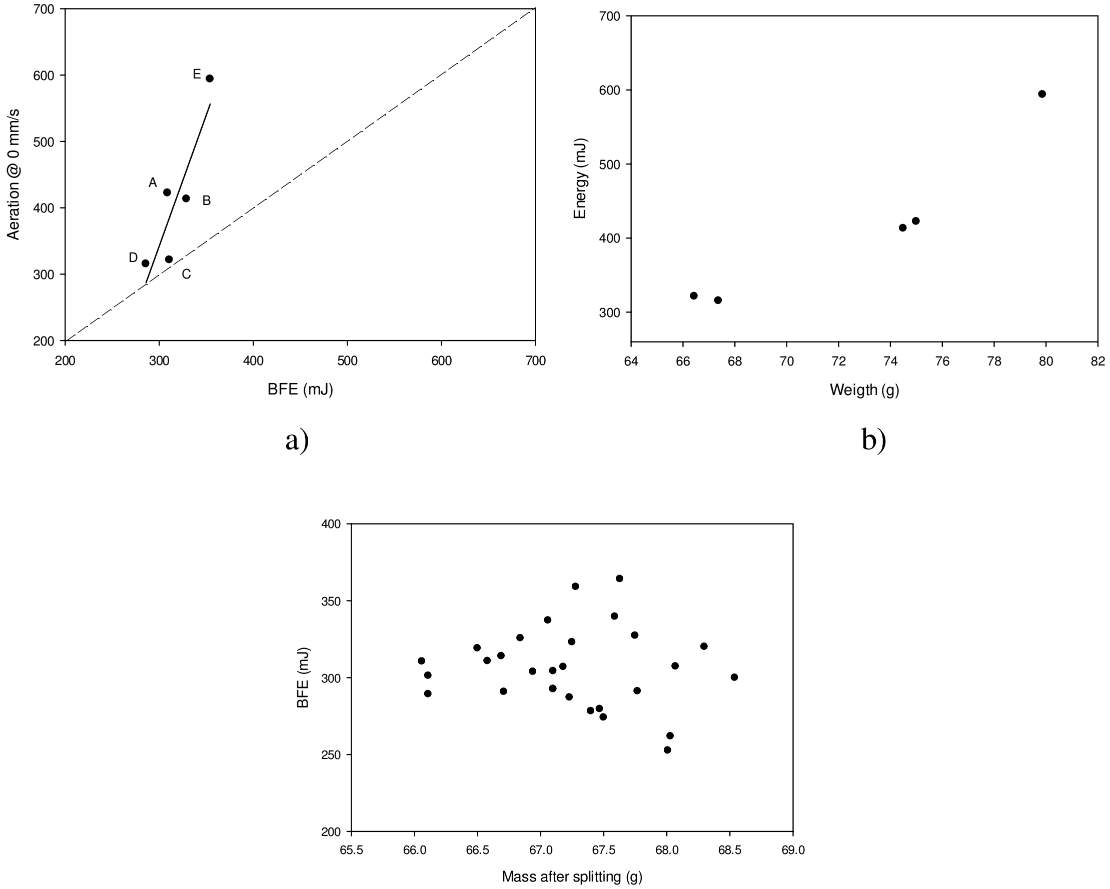
4. Discussion

The results show that there were variations during the measurement of the flow energies. The variations provided for each data point on the reproducibility tests were calculated on only three replicates and the reported values should only be used as an indication of the variability. Deviations were, however, also calculated on the entire BFE data set and the trend observed was the same. Indeed, when the BFE standard deviations is calculated with the entire population (30 data points coming from the 3 replicates in 5 laboratories measured in 2 rounds), the average BFE measured is 305 ± 26 mJ (with Bessel's correction) or ± 25 mJ (stdev P, without corrections). These values (with and without Bessel's corrections) represent 8% relative standard deviations and corresponds to the values reported in Table 1 for round 1.

Variations have already been observed by other researchers. S.V.Søgaard et al. [19] investigated the reproducibility of the measurement of the flowability of microcrystalline cellulose and anhydrous lactose using a powder rheometer and observed that the measurements of the basic flow energy of anhydrous lactose were reproducible (relative standard deviation of 3.6 %) while the variations obtained with the microcrystalline cellulose were significant (relative standard deviation of 13.2 %). The authors suggested that the variations observed were most likely not associated with environmental conditions as all tests were conducted under controlled atmosphere (50 % RH ± 5 % RH and 21 °C ± 1 °C). Besides, the authors considered that electrostatic forces were not responsible for the variations observed as the amount of charge decreases with increasing humidity and were estimated as being barely present at 50 % RH [20]. J. Whiting [21] investigated the repeatability of the measurement of stainless steel powder (17-4 SS, AM grade) also using a powder rheometer. Significant variations (540 mJ to 1010 mJ) were observed, even if the testing was conducted on the exact same sample of powder (basic flow energy ranging from 650 mJ to 900 mJ with the exact same sample).

While variations were observed within each lab (BFE relative standard variations between 2.1 to 3.6%), the inter-laboratory variations appear to be larger in the present investigation (8%). These variations are, however, smaller than those observed with a Hall flowmeter in a proficiency test program (19 laboratories) using similar powders (relative standard deviations of 18%) [17].

Comparison between the flow energy measured during the aeration test at zero air velocity and the BFE values suggests there is a correlation between the flow energy measured during the two experiments (Figure 15a). Figure 15b shows that the deviation comes essentially from the mass used during the tests. Indeed, analysis of the procedures indicated that not all laboratories used the same mass for these tests and the variation of the mass significantly affected the results. An analysis of the BFE vs mass for all tests (Figure 15c) shows that the variation of the mass during those tests was relatively small (66.7 to 68.5 g) and could not explain the variations of BFE observed during round 1 and 2.



a) b)

c)

Figure 15: Correlation between a) the flow energy measured during the aeration test at zero air velocity and BFE; b) the mass used for the aeration test and the energy measured at v=0 mm/sec during the aeration tests and c) the BFEs and mass used in the stability tests (after splitting procedure).

The variations observed by S.V.Søgaard et al. [19] were dependant on the material and not associated with the variation of environmental conditions such as temperature and humidity nor electrostatic forces. In a similar way, the present investigation showed there is no clear correlation between the flow energy and humidity level measured in the laboratory during the tests. While the humidity was not controlled in the different laboratories (measured humidity ranged between 13 % RH and 58 % RH), there is no correlation between the humidity and the flow energy measured (Figure 16).

While it is recognized that humidity does affect the flowability of fine Ti6Al4V powders [22], the sensitivity of the stability tests (and BFE) to humidity seems to be minimal, at least for the powder and conditions used in this study. In fact, previous tests conducted on the impact of humidity (0 % RH to 44 % RH) on the energy flow energy of Ti6Al4V (smaller than 45µm particles as identified by the manufacturer) showed the limited effect of the humidity in the laboratory on the flow energy measured with the rheometer [22]. This hypothesis is consistent with observations from J. Whiting [21], who did not observe the effect of drying (24 h at 100 °C) and exposure to moisture (powder left out in lab air at 45 % RH for >100 h) on the variability of tests measured on a 17-4 SS AM powder.

Modification of interparticle forces caused by humidity adsorption are potentially too small to be measured using the BFE index, at least for the material and conditions investigated in this study. Indeed, BFE is measured while the blade is moving downward and the powder is confined and pushed toward the bottom of the vessel. Under those conditions, the stresses are probably much higher than the variations of stresses that could be caused by the atmospheric adsorption of moisture. In addition, conditioning of the powder was most likely not constant from one test to the other and moisture adsorption was probably not perfectly correlated with the amount of humidity in the laboratory. Indeed, moisture adsorption may take time and vary more or less rapidly depending on powder storage, exposure to the laboratory environment and set-up of the experiment. Consequently, additional tests should be done to confirm the effect of humidity on the rheology of the powders and validate how it may impact the variability of the results.

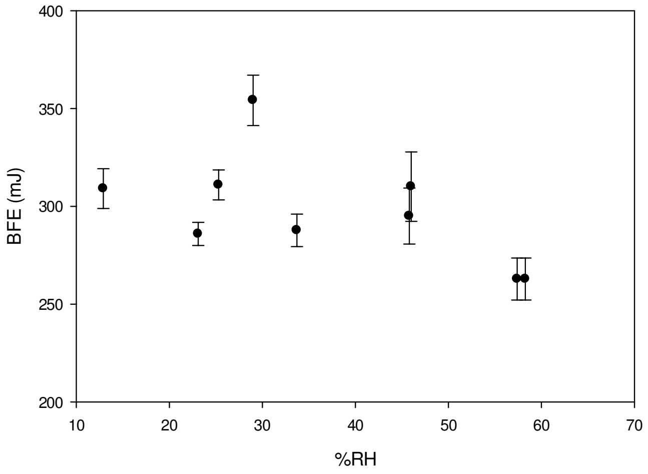


Figure 16: Effect of relative humidity on the BFE (averages and standard deviations) measured during tests conducted in round 1 and 2.

Electrostatic charging of the powder during production, shipping, handling and/or testing could potentially affect the rheology of the powder and influence the variability of the measurements. A previous study showed that charging Ti6Al4V powder by mixing in a polyethylene bottle affects the Carney flowability of the powder [22]. Unfortunately, the electrostatic potential of the powder before and after the tests was not evaluated in the present study and it was not possible to correlate the variations observed with charging. As the energy appeared to be relatively stable during the stability tests (Figures 5, 13), particles modification and charging probably did not significantly impact the measurements of the flow energy during the tests. As charging was previously observed in other experiments [23], it is believed that the tests were probably not long enough with the materials and conditions used in the present study to observe a significant charging, sufficient to impact the flow energy.

A source of variation to consider is the handling of the powder during the test (may impact humidity adsorption or desorption, charging, segregation, packing of the particles…). The development of a more direct procedure to make the measurements more reproducible from laboratory to laboratory was not, however, effective to reduce the variations between the different laboratories. Indeed, the variations observed when the laboratories were using their internal procedures (as long as they were conforming to the general guidelines of the rheometer manufacturer) are of the same order of magnitude as those obtained when each step of the measurement was fixed.

While the variations remained more or less unchanged, the average values obtained in the second sets of experiments (i.e., round 2), are in most cases smaller. The largest variations observed from round 1 and 2 came from two labs that had performed an instrument calibration (the instrument failed the calibration audit and equipment set-up had to be adjusted according to the manufacturer recommendations). Beside calibration, it would be valuable to develop reference materials that could be used to validate that the equipment is well calibrated and measurements are stable and reliable.

It is worth mentioning that previous investigations showed that the flow energy can be significantly different when the powder is characterized in cylinders made from different materials. Indeed, tests conducted on Ti6Al4V powder (less than 45 µm in size as identified by the powder manufacturer) measured in glass, stainless steel and aluminum cylinders led to very different results (BFE ranging from 259 mJ to 380 mJ) [22]. As the stresses measured in [22] were relatively stable from test to test, it is believed that the differences observed were not coming from the charging of the powder but from the interaction between the powder and the surface of the cylinder (i.e., effect of composition and surface finish). The effect is not surprising considering that the clearance between the rotating stainless steel blade (23.5 mm diameter) in the cylinder (25 mm diameter) is relatively small (750 µm) and the interaction between the powder and the surface of the cylinder may represent a significant portion of the stress measured. J.Whiting [24] observed a similar trend with 17-4 SS powder and noticed that the variations were larger with a glass cylinder when compared with results obtained with an aluminum cylinder (the difference was in this case associated with charging). In the same study, the effect of cleaning the cylinder (warm tap water, doused in isopropanol and dried with compressed air) on the results was observed, also suggesting that the interaction between the powder and the cylinder was significant. It is worth mentioning that the impact of powder-flowmeter surface interaction has been observed with other test methods, such as Hall and Carney flowmeters.

Figure 17 presenting the BFE as a function of the particle size (i.e., D50 or 50th percentile by volume measured on the different samples) shows that the difference of size distribution is very small from one sample to the other (data collected for samples of round 2 characterized by labs A, D and E), and there is no correlation between the BFE and the particle size (similar observations were obtained when comparing the D10 and D90 results). This suggests that the variations observed cannot be directly linked to variations of particle size from sample to sample (or segregation during handling or shipping).

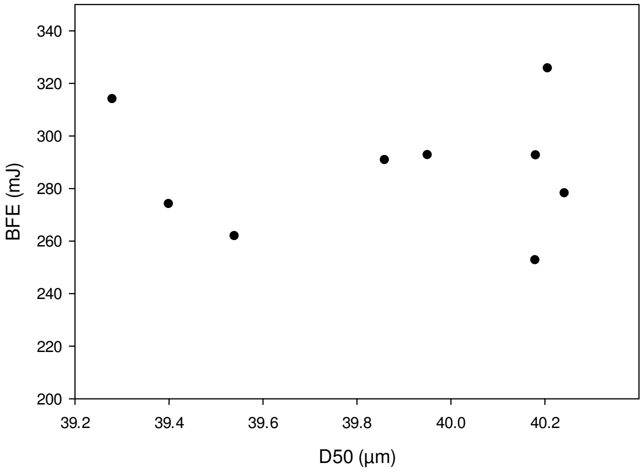


Figure 17: BFE as a function of the particle size (D50). Measurements were done on a subsample (obtained by riffling) of the samples tested in the rheometer.

In order to eliminate the potential effect of equipment calibration, specimens (2) were characterized in two of the participating laboratories to see if similar results could be obtained when the exact same specimens are characterized in different laboratories. Samples with the lowest (L) and highest (H) values in round 2 were exchanged between laboratories A and E. BFE results presented in Figure 18 show that while Lab A obtained lower BFE values for both specimens, the difference was relatively small for sample E. As the sample was the same, the variations was most likely not coming from differences of particle size. It is not, however, possible to confirm if the variations observed are coming from differences of procedures or the modification of the powder during shipping and handling (i.e. humidity, charging). As the difference is not systematically the same between the two laboratories, the differences are probably not coming for the calibration of the instruments. Additional tests should, however, be conducted with more samples to investigate further the sources of the variations observed.

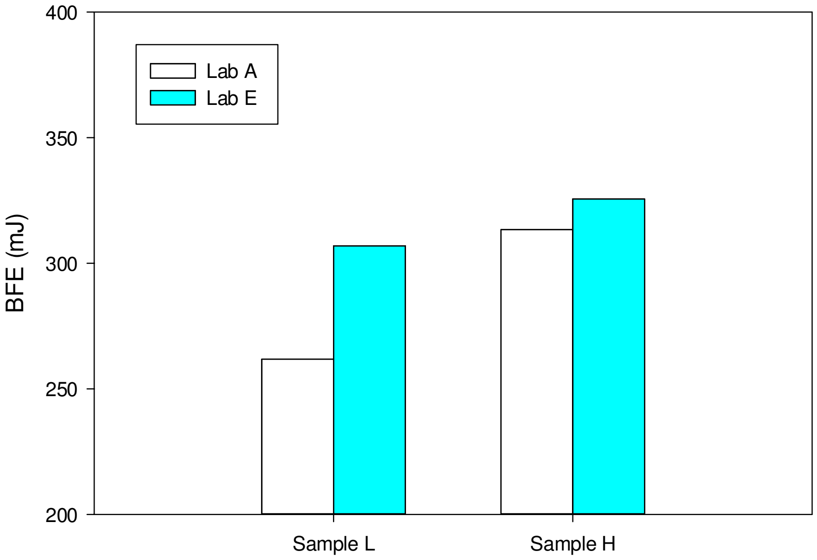


Figure 18: BFE measured in laboratory A and E with the samples that provided the lowest (L) and highest (H) BFE in round 2 (single specimen).

Attempts to correlate the differences of powder behaviour with the measurement procedure, powder sampling, charging or humidity did not enable to isolate the sources of the variations observed. Among the sources that may potentially be responsible for the variation of the results, the stability of the machine needs to be further investigated. While calibration tests done by J. Whiting [21] showed no substantial differences in measured torque or normal force (< 0.5 % change), an investigation of the calibration of the equipment in different laboratories could be done to confirm that the variations are not coming from differences in the rheometer or inadequate calibration procedures. Additional tests should also be done to further investigate the effect of particle characteristics on the flow energy. The evolution of the properties of the powder during shipping, storage, handling and testing as well as the impact of the operators on the results should also be further investigated.

5. Conclusions

Different flow tests (basic flow energy, sensitivity to blade speed, effect of powder aeration, permeability) were conducted using a powder rheometer. All tests were conducted with the same powder (different samples of the same batch that was divided by splitting into different bottles) shipped to different laboratories. Differences were observed in the powder rheology metrics. These observations were consistent with observations made in other investigations on the stability of the results obtained with a powder rheometer or flowmeter. Differences observed during the aeration tests were more important due to differences in the procedures in the different laboratories (i.e. mass of the powder used) while the variations observed during the permeability tests were minimal.

PDF-XChange Product Click to buy NOW! www.tracker-software.com

While variations have been observed in other inter and intra-laboratories studies using other flow measurement techniques, it would be important to understand the source of the variations and find ways to minimize them. Different sources were investigated (powder sampling, measurement procedures, humidity) in the present study but the variations could not be correlated to a single source. Among the parameters that need to be further investigated is the stability and calibration of the equipment as well as the effect of the blades and cylinders on the measurements. The effect of the modifications of the properties of the powder during shipping, handling, storage and testing also needs to be further investigated.

6. Acknowledgements

The authors would like to acknowledge the contribution of Shirley Mercier and Roger Pelletier for setting up the tests and the characterisation at the National Research Council Canada (NRC) and their support in the preparation of the test protocol. The authors would also like to thank Vladimir Brailovski from the École de technologie Supérieure (ETS), Ehsan Toyserkani from the University of Waterloo for their support of this work and Ed Garboczi and Shawn Moylan from the National Institute of Standards and Technology (NIST) for suggestions in the manuscript.

7. References

[1] J. K. Prescott, R.A. Barnum, On Powder Flowability, Pharmaceutical Technology, October (2000) 60-84.

[2] A. Castellanos, J. M. Valverde, A. T. Pérez, A. Ramos, and P. K. Watson, Flow Regimes in Fine Cohesive Powders, Physical Review Letters, 82, 6, (1999), 1156-59. https://doi.org/10.1103/PhysRevLett.82.1156

[3] AMSC 18-001, PRELIMINARY FINAL DRAFT AMSC ROADMAP VERSION 2.0 4/6/18, America Makes & ANSI AMSC Standardization Roadmap for Additive Manufacturing, (2018).

[4] MPIF 03, Method for Determination of Flow Rate of Free-Flowing Metal Powders Using Hall Apparatus, Standard test methods for metal powder and powder metallurgy products, Metal Powder Industries Federation, (2016).

[5] ASTM B213-17, Standard Test Methods for Flow Rate of Metal Powders Using the Hall Flowmeter Funnel, ASTM International, West Conshohocken, PA, 2017, www.astm.org, http://dx.doi.org/10.1520/B0213-17

[6] ISO 4490 Metallic powders – Determination of flow rate by means of a calibrated funnel

[7] ASTM D7481-18, Standard Test Methods for Determining Loose and Tapped Bulk Densities of Powders using a Graduated Cylinder, ASTM International, West Conshohocken, PA, 2018, www.astm.org, http://dx.doi.org/10.1520/D7481-18

[8] B.H.Kaye, Characterizing the Flowability of a Powder Using the Concepts of Fractal Geometry and Chaos Theory, Particle & Particle Systems Characterization, 14 (1997) 53-66.

[**9**] B.H Kaye., J. Gratton-Liimatainen, N. Faddis, Studying the Avalanching Behaviour of a Powder in a Rotating Disc, Particle & Particle Systems Characterization, 12,5 (1995) 232-236.

[10] Mercury Scientific Inc., Flowability analysis with the revolution, http://www.mercuryscientific.com/instruments/flowability-analysis-revolution, (accessed 25 July 2019)

[11] GranuTools, GranuDrum- Granular material flow analyzer-Powder rheometer, https://granutools.com/products/granudrum/, (accessed 25 July 2019)

[12] ASTM D6467-13e1, Standard Test Method for Torsional Ring Shear Test to Determine Drained Residual Shear Strength of Cohesive Soils, ASTM International, West Conshohocken, PA, 2013, www.astm.org,
http://dx.doi.org/10.1520/D6467-13E01

[13] ASTM D3080 / D3080M-11, Standard Test Method for Direct Shear Test of Soils Under Consolidated Drained Conditions, ASTM International, West Conshohocken, PA, 2011, www.astm.org, http://dx.doi.org/10.1520/D3080_D3080M-11

[14] Freeman technology, Powder Flow Testing with the FT4 Powder Rheometer. https://www.freemantech.co.uk/_powders/ft4-powder-rheometer-universal-powder-tester, (accessed 25 July 2019)

[15]Anton Paar, Rheometer, https://www.anton-paar.com/us-en/products/group/rheometer/, (accessed 25 July 2019)

[16] Controls Group Inc., Bromhead ring shear apparatus, http://m.controls-group.com/eng/products/rock-mechanics-testing-equipment-testing-equipment-testing-equipment/bromhead-ring-shear-apparatus

[17] Committee B09 Proficiency Test Program Additive Manufacturing Powder Metallurgy Sample ID: AMPM1804 April 2018, Report Issue Date: September 13, 2018. ASTM International, West Conshohocken, PA

[18] https://www.freemantech.co.uk/learn/product-brochures.

[19] S.V.Søgaard, M.Allesø, J.Garnaes, S.Baldursdottir,, J.Rantanen, Development of a Reproductible Powder Charactrization Method Using a Powder Rheometer, Annual Transactions of the Nordic Rheology Society, 20 (2012) 239-245.

[20] G. Léonard, N. Abatzoglou, Lubrication of pharmaceutical powder/wall interfaces and electrostatic effects, Powder Technology, 208 (2011) 54–62.

[21] J. Whiting, Repeatability of the FT4 Rheometer Evaluating AM Powder, presented at Additive Manufacturing with Powder Metallurgy (AMPM), June 13-15, 2017 MPIF, Las Vegas, USA.

[22] L.P.Lefebvre, F.Bernier, R.Pelletier, Flowability of powders: Adapting the measurements to AM requirements, presented at Additive Manufacturing with Powder Metallurgy (AMPM), June 17–20, 2018 MPIF, San Antonio, USA.

[23] L.P.Lefebvre, F.Bernier, N.Orsoni-Wiemer, C.Charbonneau, B.Alchikh-Sulaiman, S.Yue: Rheology of Powders: Assessing the Robustness and Impact of Humidity, Tribocharging, Particle Size and Composition, submitted to Powder Metallurgy.

[24] J.G.Whiting, Key Parameters and Issues: Effects of Particle Size, Particle Size Distribution, and Particle Morphology, presented at the HPC4Mfg workshop on Modeling of Powder Dynamics in Metal Additive Manufacturing, August 10-11th, Austin, Texas 2017.