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REPEATABILITY AND SENSITIVITY OF A ROTATING DRUM METHOD FOR RHEOLOGICAL CHARACTERIZATION OF STAINLESS STEEL POWDERS USED FOR ADDITIVE MANUFACTURING

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Abstract

There remains a need for rheological characterization of metal powder used for powder-spreading-based additive manufacturing (AM). Novel powder rheometers introduced to the commercial market for this purpose must be rigorously evaluated for repeatability and sensitivity before widespread adoption for predicting AM powder performance. The work presented here focuses on the quantification of the repeatability and sensitivity of a commercially available rotating drum powder rheometer for testing metal AM powder. This assessment is accomplished via a set of tests that include the following independent variables: cleaning method, the mass of the sample, particle size distribution, material, and hysteresis.

Introduction

Metal-based additive manufacturing (AM) is capable of producing layer-by-layer, complex metal parts used for biomedical, aerospace, and other applications. However, the prerequisite for widespread acceptance of any manufacturing technique is consistency and reliability of the process and products. It is generally understood that variability in feedstock materials (e.g., metal powders) will lead to variability in final part performance. Obviously, the characterization of metal powder and determining its consistency are extremely important for AM [1].

Metal powders used in AM are often characterized by their size, morphology, density, chemical composition, flow rate, and thermal properties [1]. Various metal powder characterization techniques for the AM industry are defined in ASTM F3049 [2]. There exist several commercial instruments for powder characterization for each of the powder aspects. Typical powder characterization requirements and the corresponding instruments include (i) particle dimensional analysis (e.g., sieving, laser diffraction, dynamic imaging analysis, scanning electron microscopy, X-ray computed tomography), (ii) density analysis (e.g., apparent density analyzers like Hall funnel and Scott Volumeter, tapped density analyzer, skeletal density analyzer), (iii) chemical

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composition analysis (e.g., inductively coupled plasma mass spectrometry, energy-dispersive X-ray spectroscopy, X-ray fluorescence spectroscopy, etc.), and (iv) flow analysis (e.g., rotating drum powder rheometer, rotating blade powder rheometer, angle of repose method).

Here, we would like to emphasize that the inner workings and, often, the measurement uncertainty of most of the commercial methods are not known to the users. It is not uncommon to have contradictory results obtained for the same powder from different methods [3-4]. Moreover, variability in the instruments that are based on the same measurement principle exists. Therefore, it becomes necessary to evaluate these instruments carefully before adopting them for metal powder characterization.

In AM, metal powder flow analysis is considered valuable information for predicting powder spreadability in powder bed fusion (PBF) machines. Powder flow analysis using a Hall funnel and determining the angle-of-repose usually gives some indication of the powder flowability. However, for PBF applications, rotating drum or rotating blade powder rheology methods are frequently used [5-7] because they provide, arguably, more relevant powder assessment capabilities [8].

The rotating drum powder rheology method was the focus of this work. As shown in Figure 1, a rotating drum rheometer used for evaluating the flowability of the metal powders used in the AM is basically a horizontal cylinder with two glass (or polycarbonate) side walls. The sides are coated with a conductive coating to avoid triboelectric charging. Usually, the drum is approximately 50 % by volume filled with the powder for testing. The drum is forced to rotate by a drive roller around its axis at some angular velocity causing the powder to avalanche and flow inside the drum. The drum is backlit, and the images of the powder-filled drum are captured using a camera. The powder-filled area of the drum appears dark and the rest of the air-filled drum appears bright. The contrast and brightness are adjusted so that the powder and the air interface are detected by an edge-detection algorithm. This interface has been characterized and categorized in a variety of ways. One of the more popular techniques involves measuring the angle between a best-fit line to this interface and horizontal. This is often termed the dynamic angle of repose. Elsewhere, the variability of this interface has been compared to its average position to infer what is termed the cohesive index [5], though the exact definition is not provided.

In this work, we have evaluated the general repeatability and sensitivity of a commercially available rotating drum powder rheometer to a variety of independent variables including particle size distribution (PSD), sample mass, alloy, and the cleaning method.

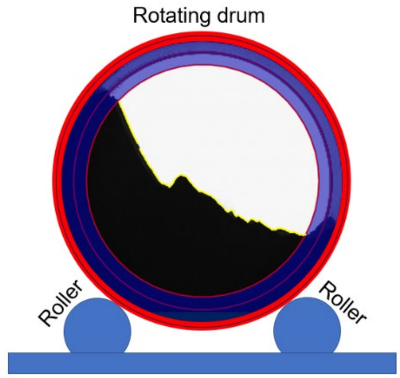


Figure 1: The schematic of the front-view of a rotating drum powder rheometer. The powder-filled drum can be rotated clockwise if rollers rotate anti-clockwise and vice versa.

Materials and Methods

In this study, four different stainless steel powders were used: one commercially available laser PBF 17-4 stainless steel powder (vendor specified median particle size range: 36 µm to 44 µm) and three different types of 17-4 stainless-steel powders [9, 10] with customized PSDs: 'Coarse' powder of median equivalent circular area diameter (ECAD) = 38.4 µm, 'Medium' powder of median ECAD = 34.4 µm, and 'Fine' powder of median ECAD = 14.4 µm. The PSDs of the three customized powders were measured using dynamic image analysis at NIST. All powders used in this study were in an as-received condition.

The rotating drum used in this study typically requires 55 mL of powder for testing, but to avoid variability introduced via varying apparent density, in this study, all powder samples were measured by mass and not by volume. The mass corresponding to an apparent density of 55 mL was found to be 221.1 g for the commercially available stainless steel powder. The sample preparation was done following ASTM B215-15 [11]. The lab humidity [relative humidity (RH)] during these experiments was 35 % ± 5 % and the temperature was 22.5°C.

Calibration and adjusting of the contrast and brightness for the instrument were done for each powder according to the user manual. After loading the powder into the drum, powder-conditioning was done for each sample by rotating the drum at 15 RPM for 60 sec. After two minutes of wait time (i.e., without the drum rotation for the sample to rest), the tests were performed. For each sample, a rotational velocity sequence was investigated using the following rotational speeds: 2 RPM, 4 RPM, 6 RPM, 8 RPM, 10 RPM, 20 RPM, and 30 RPM. This progression through these rotational speeds is considered one 'test'. As explained earlier, the powder-air interface can be characterized in many ways. This particular rotating drum powder rheometer employs the metrics of the dynamic angle of repose (˚) and the cohesive index (unitless, proprietary metric) primarily, but also provides data on volume fractions by evaluating the regions of the image with and without powder. To avoid the edge effects from powder adhering to the perimeter of the drum, the radially-outermost portion of the image is not considered for calculating these metrics. This image cropping removes a fraction of the image area which is operator-defined in terms of an area percent. For this work, 10 % of the area was removed for all results. For brevity's sake, for the remainder of this manuscript, the cohesion index will be referred to as "cohesion" and the dynamic angle of repose as "angle."

Each sample was also tested six consecutive times without opening the drum. The idle time between two repeat tests was < 10 s. Repetitive testing of the same sample is generally not recommended due to the often-inconsistent results from the evolving system behavior during testing. The potential sources of inconsistency are listed below in the test list under item 5), monotonicity. Nonetheless, these tests were conducted in an effort to understand the evolution of the behavior of the powder and to characterize the trends seen. This may provide answers to the following questions: do the trends of the measured angle or cohesion plateau, and at what point does the behavior change significantly?

The following experiments were performed:

1) Comparison of inter-sample variability versus repeat testing variability: For evaluating the repeatability and the effect of repetitive testing, the commercially available laser PBF 17-4 stainless steel powder was used. Six samples were used. Each sample was tested six times repetitively (i.e., without removing it from the drum). The first tests from each of the six samples were compared with the data from all repeats (i.e., six tests from each sample). This was primarily to understand the variability in repeat testing compared to first tests of the samples only.

2) Effect of cleaning methods: there are two typical ways the drum can be cleaned. The first is without using isopropanol or ethyl alcohol (dry cleaning) and the second is using isopropanol or ethyl alcohol (wet cleaning). In the dry cleaning method, the drum cell was cleaned by using a small craft paint brush only. In the wet cleaning method, after cleaning the drum using the brush, the drum was also cleaned using ethyl alcohol (190 proof) and lint-free tissue papers. The drum was allowed to dry for 15 min to ensure all ethyl alcohol had evaporated. To evaluate the necessity of the wet-cleaning method, seven samples of the commercially available laser PBF 17-4 stainless steel powder were tested, six for the dry-cleaning method and one sample for the wet-cleaning method.

3) Effect of particle size distributions: the Coarse, Medium, and Fine powders were tested and the results were compared. These powders had been previously characterized in previous work [9].

4) Effect of sample mass variation: To understand the effect (if any) of different sample masses, the control sample size of 221.1 g was compared to four different sample sizes with masses of 90 %, 95 %, 105 %, and 110 % of the nominal, control mass. For this, the commercially available laser PBF 17-4 stainless steel powder was used.

5) Evaluation of the presence of monotonicity: evaluated by repetitive testing of the same material. For this, the commercially available laser PBF 17-4 stainless steel powder was used. No specific test is conducted, but rather the data from a series of repeat tests was evaluated for a monotonic tendency (i.e., uni-directional increase or decrease in the magnitude of angle or cohesion). There are a variety of sources that can produce a non-monotonic nature including coating of the drum's walls, triboelectric charging, moisture sorption or desorption, and changes to the device (e.g., friction-inducing warming of components). This experiment may not provide a root cause analysis, but only identify the presence and quantify the magnitude of monotonicity.

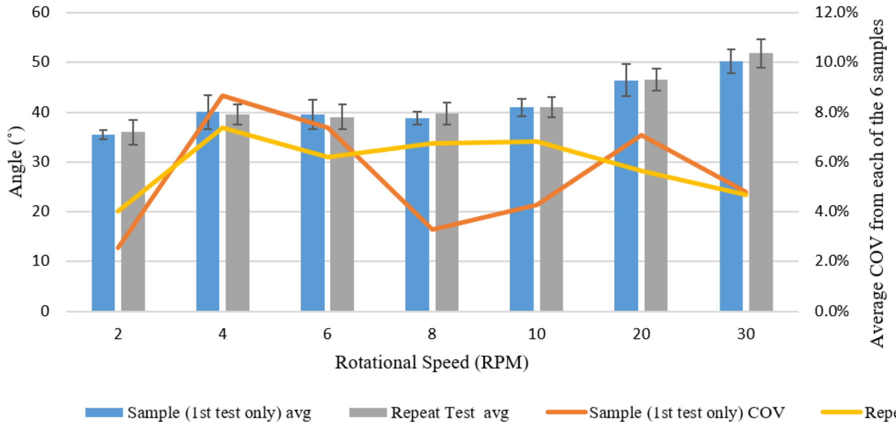
6) Evaluation of the presence of hysteresis: hysteresis is the dependence of some characteristic of a system on the history of the system. In order to detect the presence of hysteresis, one of the customized PSD powders (Fine), previously referenced in item 3), was tested using the normally prescribed, monotonically increasing rotational speeds for four tests. On the fifth test, the same rotational speeds were used, but in the reverse order. This testing scheme can provide insight into whether the stresses imparted on the powder during previous rotations will affect future results. Here, hysteresis is said to be present if significant difference is apparent between the 'normal' ascending speed data and the descending speed data.

Results and Discussion

This section is organized by the independent variables each of the experiments had employed, and is partially chronologically organized. For instance, since the operators wanted to know if dry cleaning was sufficient and did not introduce significant differences or variability compared to a wet cleaning, these tests were conducted first so that they could guide the remaining experiments.

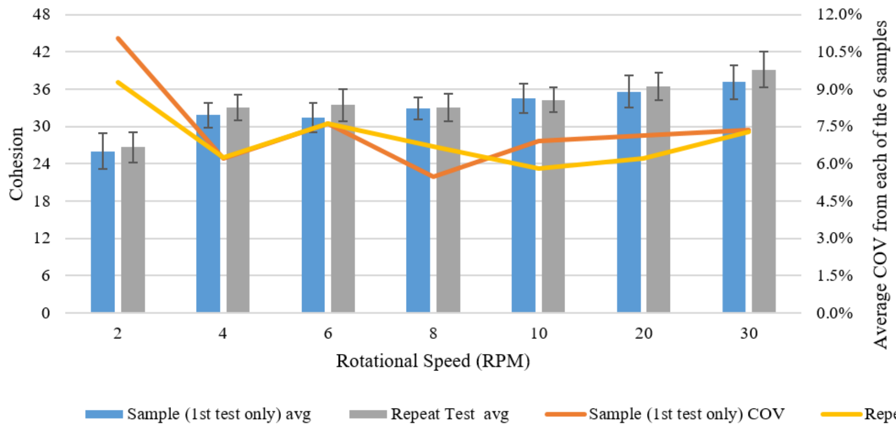
1) Comparison of Inter-sample Variability Versus Repeat Testing Variability

This section serves to quantify the general repeatability of the method given the aforementioned settings and to compare inter-sample variability and the variability present when testing the same sample repeatedly. The 'sample' statistics are taken only from the first test of each sample while the 'repeat test' statistics are taken from all data (i.e., all six samples and all six repeats). First, as shown in Figure 2, the magnitudes, standard deviations, and coefficients of variation (COV) for cohesion and angle are plotted for both sample averages and repeat test averages for only the dry-cleaning method. Besides the slightly lower COV at 8 RPM and 10 RPM for the inter-sample results for the angle metric (Figure 2a), the difference in averages and the variability is negligible in that the error bars, which represent ± one standard deviation, overlap the mean values. Overall, the COVs remain between 2.5 % and 8.7 % for all angle data and between 5.4 % and 7.6 % except for 2 RPM for the cohesion metric. The 2 RPM cohesion data have COVs of 9.3 % and 11.1 % for the repeat test and the sample COV respectively, which is significantly larger than the faster rotational speeds. For some powders, it is useful to have a repeat testing to produce larger data sets since the most time-consuming and operator-intensive part of these tests is the sample swap, which requires cleaning to ensure minimal cross-contamination. More work is needed to confirm this finding for other powders as certain powders are more affected by triboelectric charging and are more prone to coating the drum's walls.



Speed (RPM)	Repeat Test COV	Sample COV
2	4.0%	2.6%
4	7.4%	8.6%
6	6.2%	7.4%
8	6.7%	3.3%
10	6.8%	4.3%
20	5.6%	7.0%
30	4.7%	4.8%

(a)



Speed (RPM)	Repeat Test COV	Sample COV
2	9.3%	11.1%
4	6.2%	6.2%
6	7.6%	7.6%
8	6.7%	5.5%
10	5.8%	6.9%
20	6.2%	7.1%
30	7.3%	7.4%

(b)

Figure 2: (a) Angle and (b) cohesion index data with variability (COV) between repeat tests and between samples. Error bars represent ± one standard deviation. The "Sample avg" and corresponding COV are computed using the data from the first test of each sample, while the "Repeat Test avg" and corresponding COV represent the statistics from the average of the six samples.

2) Effect of Cleaning Method

This section serves to evaluate the two cleaning methods. Figure 3 presents a comparison of the cleaning method employed and also a comparison of the inter-sample variability with repeat test variability. While Figures 3(a) and (b), which only use data from the first test of each sample, display a general agreement between the wet cleaning data and dry cleaning data, when the repeat testing data is included the values align more closely. In fact, as shown in Figures 3 (c) and (d), the error bars of each cleaning method overlap the average values from the other for all but one rotational speed and metric (angle and cohesion) combination (cohesion data at 30 RPM). If there was a considerable difference (non-overlapping error bars) it would indicate the method of cleaning is affecting the measurement process. This indicates that the cleaning method does not appear to alter the results, therefore for the remaining tests, only the dry cleaning technique is employed.

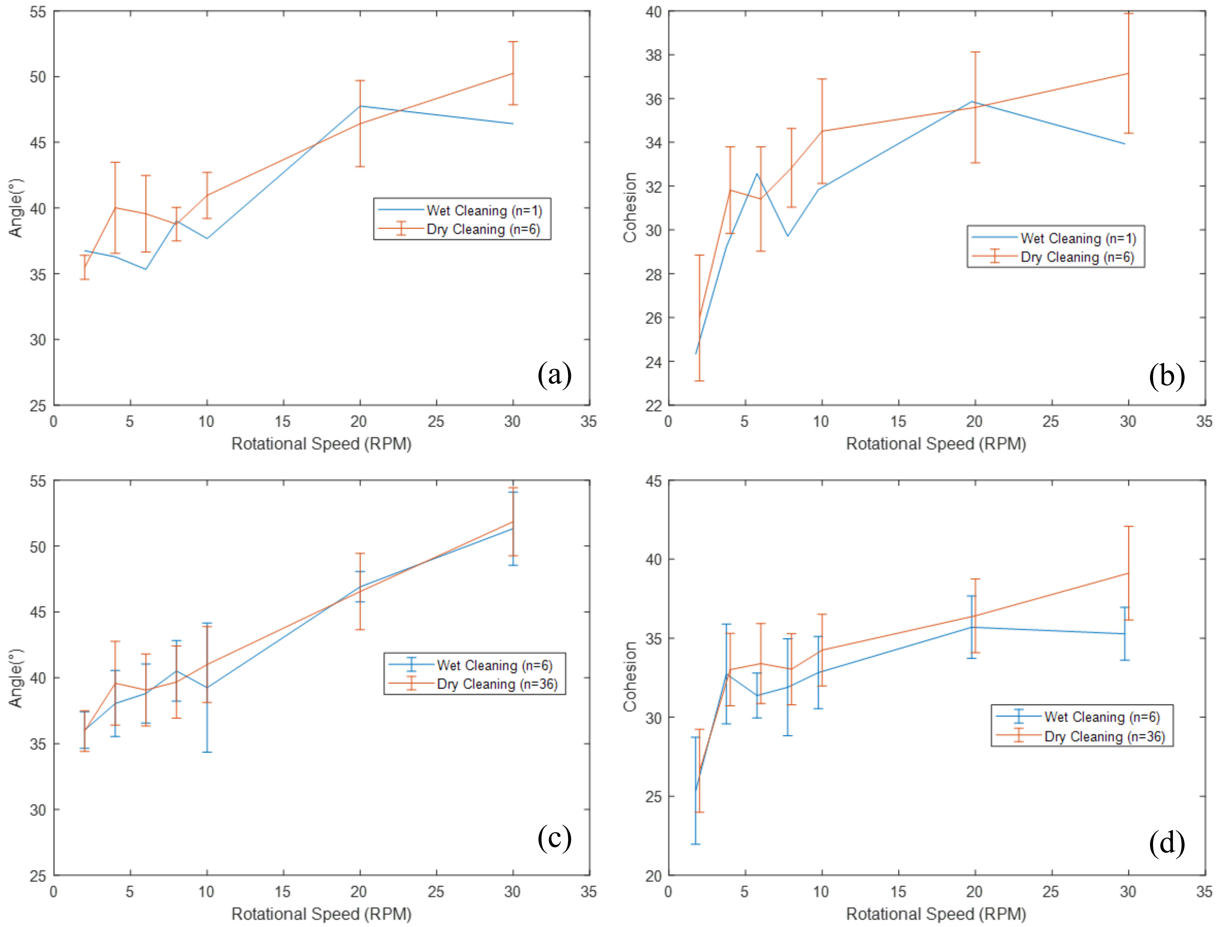


Figure 3: (a) and (b) contain results from the angle and cohesion results of only the first test on each sample, while (c) and (d) include the repeat testing data from each sample. Error bars, where applicable, represent ± one standard deviation of the data. Note: the number of data points (n) are included in each of the legends and that the wet cleaning data in (b) and (d) is shifted by -0.25 RPM along to the X-axis to aid in visualization.

3) Effect of PSD

Figure 4 contains data on both the angle (Figure 4a) and cohesion (Figure 4b) metrics and error bars representing the standard deviation of six repeat tests from each of the customized PSD powders. For angle, the three materials are clearly differentiated at rotational speeds of 6 RPM and faster with both the Normal and Coarse materials showing a generally proportional relationship between angle and rotational speed. The Fine powder created the highest measured angle of the three powders, but only at 2 RPM, the lowest speed, and had an inversely proportional relationship between rotational speed and angle. The cohesion metric had similar trends, but with the Coarse and Normal powders producing negligibly different results (overlapping error bars) for all of the rotational speeds. Interestingly, as in the case of Fig. 4b (cohesion data), the Coarse and Normal powders' data are grouped closely to one another, which was also documented in the case of a rotating blade powder rheometer [9]. Also, the Fine powder produced the lowest total energy in the rotating blade powder rheometer [9] indicating the least resistance to the blade's motion. Similarly, the Fine powder shows the lowest cohesion for all rotational speeds and the lowest angle for all speeds, but 2 RPM, indicating a free-flowing powder. This disagrees with the other measurement methods employed in [9]. For instance, both the Coarse and Normal powders were able to flow through the Hall flowmeter whereas the Fine powder was not.

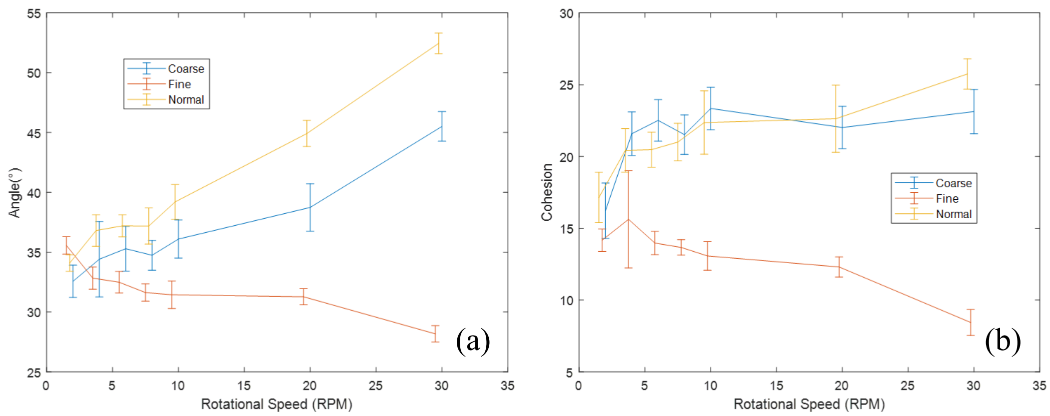


Figure 4: (a) The angle results plotted for three powders with unique PSDs and (b) the cohesion results from the same three powders. Error bars represent ± one standard deviation of the six repeats conducted on each sample. Note some of the data is shifted slightly in the +/- x-axis direction to aid in visualization.

4) Sensitivity to Change in Test Mass

The manufacturer's prescribed sampling technique employs a graduated cylinder with 20 mL increments and suggests the operator measure ≈ 55 mL. This volume corresponds to 221.1 g for stainless steel with the apparent density of 4,020 kg/m^3. So, this method has a resolution uncertainty = 10 mL, which corresponds to 40.2 g (i.e., 18.2 % of control sample mass, 221.1 g). Since it is unlikely an operator would acquire a sample corresponding to the extremes of the graduated cylinder's increments, a series of tests using ± 10 % and ± 5 % of the control sample mass of 221.1 g was used.

As seen in Figure 5, there is a clearly discernable difference between the extreme sample masses (i.e., + 10 % and – 10 % of the control mass) in the angle data (Figure 5a) at higher rotational speeds. The cohesion data (Figure 5b) provides clearly discernable trends at higher rotational speeds, though the error bars are overlapping. At the lower speeds, the data is grouped more closely, potentially indicating a negligible difference. For the ± 5 % mass deviations, the error bars (one standard deviation of six repeats) overlap the average values indicating this level of sample mass variability is acceptable for this material and at these speeds.

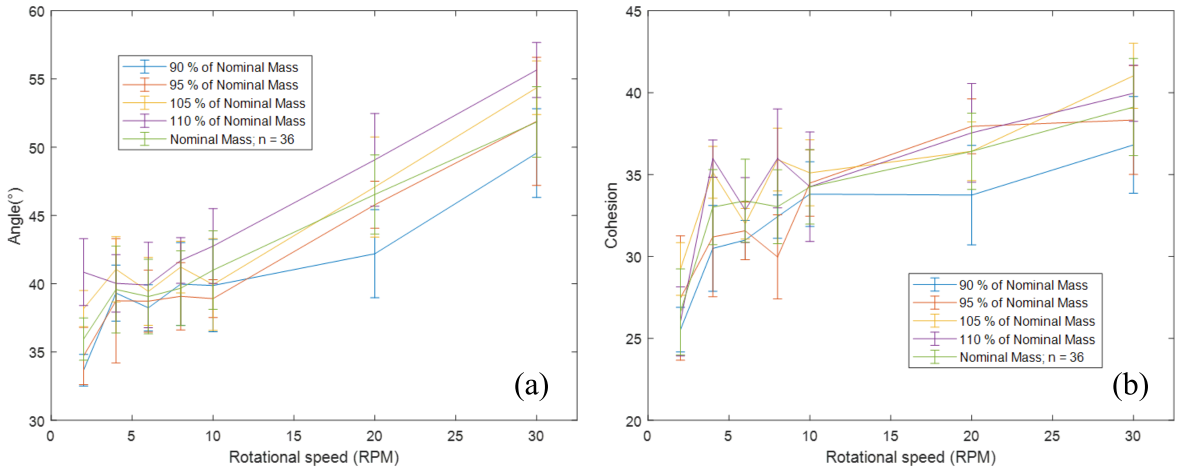


Figure 5: (a) The angle and (b) cohesion data for ± 10 % and ± 5 % of the control sample size and control sample mass. Error bars represent +/- one standard deviation of the six repeats conducted on each sample.

5) Monotonicity

To investigate the evolution of the powder's behavior given the applied stresses, six samples each tested six times repeatedly are plotted in Figure 6 along with the best fit line for each *sample* and at each rotational speed using simple linear regression. The slope of these best fit lines can provide a means to quickly visually identify the presence of monotonic nature and the averages for each metric-rotational speed combination are listed in Table 1. While some of the individual slopes are negative, as shown in Table 1, the average slopes from all six samples are all positive or zero as is the case for angle at 6 RPM. This indicates a general positive monotonic nature during the repetitive testing of one sample. While earlier, in Fig 2, no significant difference was seen in the inter-sample variance and between repeats, this deserves more attention. The presence of monotonicity indicates a change in the behavior of the powder or the device. Further work is needed to investigate whether and where these trends plateau, and whether they exist during one test (i.e., are the stresses imparted during an individual test sufficient to induce this change?). These plots also provide a visual representation of outliers or regions with large variance. While there exists some scatter, for instance, the 26th repeat at 6 RPM, there are no consistent trends in these outliers for angle, which agrees with Fig 2(a). This is indicative of a random source of variance, not a systematic error. For cohesion there seems to be a larger variance at the lowest rotational speed, 2 RPM, which agrees with the earlier listed COVs presented in Fig 2(b). This larger repeat test COV magnitude (≈ 9.3 %) reduces at 4 RPM and remains consistent (COVs between 5.8 % and 7.6 %) for the remaining rotational speeds. The high variance of the 2 RPM data is only present for the cohesion data, meaning the angle is not varying as much as the deviation in the powder-air interface (i.e., cohesive index or cohesion). It is possible this is an artifact introduced during either the acquisition or the analysis, both of which are controlled by the instrument's software and not accessible to the user. For example, depending on the sampling rate and the rate of the phenomenon, the system may not abide by the Nyquist theorem creating the potential for aliasing to be present. This could create the relatively large variance in the cohesion data.

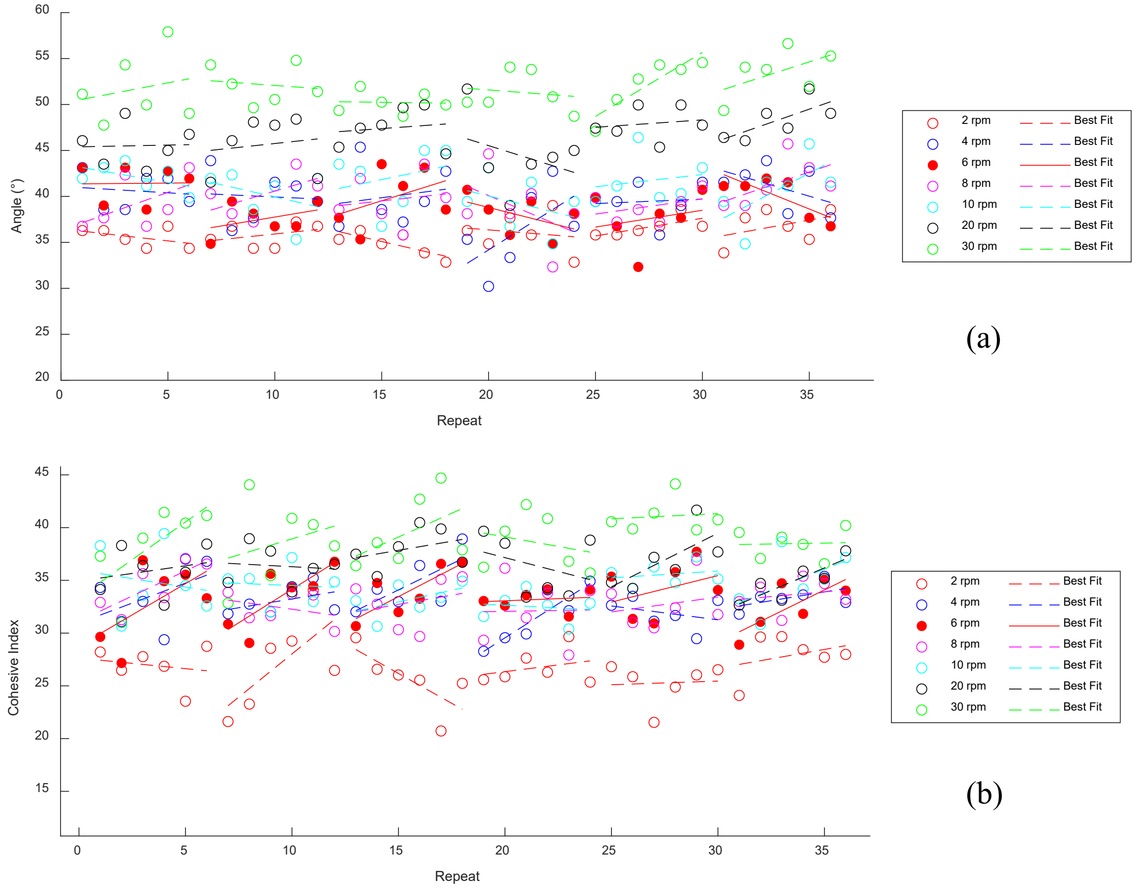


Figure 6: Individual test results plotted for each repeat of (a) angle and (b) cohesion. Best fit lines for each sample (consisting of six tests each) and at each rotational speed are calculated via a linear regression.

RPM	2	4	6	8	10	20	30
Angle (˚)	0.01	0.16	0.00	0.32	0.08	0.12	0.37
Cohesion	0.16	0.57	0.85	0.25	0.14	0.31	0.45

Table 1: Average coefficients (slopes) of best-fit lines from each of the rotational speeds from Figure 6

6) Hysteresis

Figure 7 contains the results with 'Hyst Down' referring to the fifth test done in descending RPMs. The 'Hyst Down' data shows no difference, in angle, cohesion, or standard deviation for any of the data indicating no presence of hysteresis. It is interesting that the values and standard deviations have a significant increase for the 4 RPM cohesion tests for only the 'Fine 02' and 'Fine 03' tests. This anomaly is not seen in the angle data nor at any other rotational speeds.

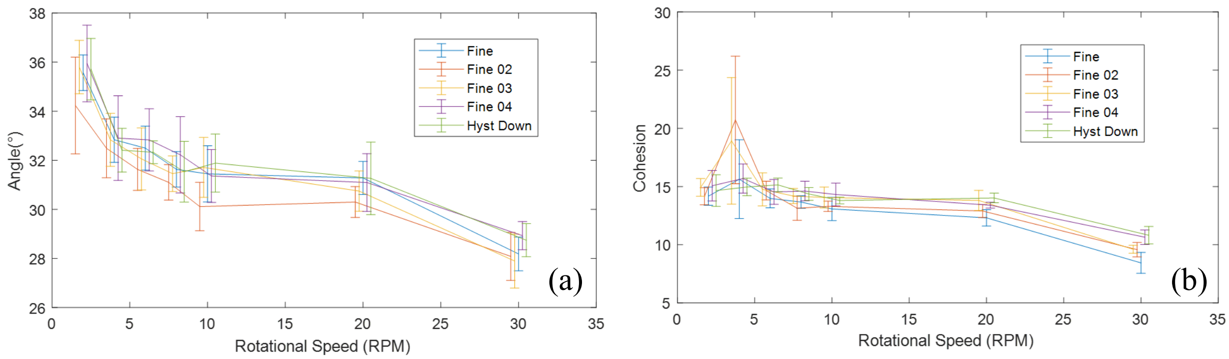


Figure 7: Hysteresis testing results for both angle, (a) and cohesion, (b). 'Hyst Down' denotes the testing completed in descending rotational speed, while all other lines represent repeat testing of the same Fine powder. Note: to aid in visualization, Fine 02 data is shifted by -0.2 RPM; Fine 03 data is shifted by -0.1 RPM; Fine 04 data is shifted by +0.1 RPM; Hysteresis Down data is shifted by +0.2 RPM.

Conclusions

In this paper, we evaluated the repeatability of the rotating drum powder rheology method for characterizing metal powders. The summary of the findings is as follows:

- A difference in both the magnitudes and trends of powders with three unique PSDs. The measured angle (i.e., dynamic angle of repose) for all three powders was discernible at the tested rotational speeds above 4 RPM. The measured cohesion of the Normal and Coarse powders was not significantly different for all but the highest rotational speed, 30 RPM, though the Fine powder was significantly different from the Normal and Coarse at all rotational speeds.
- Negligible difference in inter-sample variability and repetitive testing variability (i.e., same sample). Recall, in this paper, we consider a negligible difference as an overlap between one standard deviation and the average value of the compared results.
- Negligible difference between wet and dry cleaning suggests dry cleaning is sufficient for the materials tested.
- A difference in results between ± 10 % of the control mass and a control mass of the test sample indicating the provided sampling technique may be insufficient to ensure repeatable and reproducible testing. The ± 5 % deviations from the control sample mass did not significantly differ from the control sample mass indicating this level of sample variability is acceptable.
- Hysteresis was not detected at the tested rotational speeds and for the tested materials.

These results will impact future experiments conducted using this instrument. Dry cleaning will save a user time between tests compared to wet cleaning. We suggest controlling sample sizes using mass and ensuring all samples are within ± 5 % of the recommended mass. While it may be too much to suggest the avoidance of testing below 4 RPM, care should be taken when using low rotational speeds without better knowledge of Nyquist frequencies. Finally, more research is needed to understand the relevance of these metrics to the performance of metal powders in the actual PBF process.

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