Evaluating the sensitivity of virgin and recycled AM powders to segregation and humidity

Abstract

Powders can change their flow properties as they are handled and used. They also can become more sensitive to segregation on handling and environmental conditions. This means that a powder that has been used or recycled may change its behavior due to handling and environmental exposure more than virgin material. This behavior is evaluated by testing the flow properties of virgin and used AM powders with the Revolution Powder Analyzer before and after exposure to segregation pressure and different environmental conditions.

Introduction

AM metal powder processes such as laser and electron beam-based powder bed fusion (PBF) and binder jet printing typically require powders that flow well to function properly. Problems such as inconsistent spreadability, poor feeding, and missed layers occur when the feedstock powder either does not flow well enough or blocks feeders and spreaders. These problems can produce inconsistent parts or if severe enough can shut down the production process.

Typically, parts producers test virgin powders as received for various properties including particle size distribution, hall funnel flow and bulk density. Advanced users will also make rheological powder flow measurements by various means. A small sample taken from a larger batch is used for this testing. If the test sample has the “correct” properties then the material is used in production. The correct properties are typically determined by past experience with the powder material. After the powder is used, it is typically recycled by screening and possibly refreshed with additional virgin material. A small sample of the recycled and refreshed powder may then be tested again or may be simply loaded into the printer.

This approach generally works but sometimes a powder that has been producing parts and has tested correctly suddenly stops working well in the printer. Layers are missed or are inconsistent. Spreaders become jammed or blocked or are not fed with powder. Parts are not up to specification. Printer users search for reasons for the failures but many times they remain unexplained. The problem powder will be retested and sometimes powder property changes will be detected but an underlying reason for the change will not be determined. Other times the powder will test correctly. The typical explanations for the failure phenomena are the powder gained a static charge or humidity affected the powder or the powder segregated.

To help determine why seemingly acceptable powders start causing production problems, the flow properties of two sets of virgin and recycled samples powders were tested for sensitivity to humidity and segregation. The flow properties were then compared to determine if the flow properties of recycled samples were more or less sensitive to humidity and segregation than the virgin materials. The powders were exposed to humidity in a oven for two hours and then tested. They were then baked in a 200C dry oven for two hours to dry them. The powders were subjected to segregation stress by flowing them through three successive core flow funnels. The first portion of sample exiting the final funnel was tested. Powders were also subjected to segregation stress by shaking them in a flat pan.

The flow and charging properties of the powders were characterized with the Revolution Powder Analyzer which has been demonstrated to be very sensitive to small changes in AM powders. The analyzer has demonstrated the capability to capture differences in 316L stainless steel powders atomized by argon or nitrogen [**1**]. The argon atomized powder consistently displayed better flowability than the nitrogen atomized powders measured by flow test using the analyzer. These differences in flowability of the powders were correlated to the powder morphology, with argon atomized powder displaying higher sphericity. Differences in flowability of virgin and recycled 304L stainless steel powders were also identified using the Revolution tool [**2**]. After 7 uses, both the PSD and particle shape changed, which resulted in the different flow properties of the recycled powder. Elemental powders, Fe and Ni, were tested using the analyzer concerning their flow properties [**3**]. With different PSD and particle shape, the powders displayed different flow properties, which were successfully captured by the Revolution tool.

In this study, the sensitivity of the flow properties to humidity and segregation of two sets of powders were tested. The sets included a virgin material and a recycled sample of the material after being used in an AM printer. The two sets of powders tested displayed differences in the sensitivity of the virgin and recycled powders to segregation and humidity. In one set of samples, the virgin powder was less sensitive to segregation and humidity than the recycled material. The implication of this is that the recycled sample will change more if exposed to humidity or segregation stress than the virgin material. In the second case, the recycled material was less sensitive to segregation and humidity. Powders that are sensitive to humidity and segregation can easily become inhomogeneous. Then a poor flowing portion of powder can accumulate in one area of a printer and block flow paths or spreaders or create poor layers.

Materials

Two sets of two 316L austenitic stainless steel powder samples labeled Set A and Set B were tested. Each set consisted of a virgin sample and a recycled version of the same material. For Sample Set A, the recycled material went through two printing cycles. For Sample Set B, the recycled material went through eight printing cycles. These materials were characterized extensively in [5] and [6]. The particle size is summarized in Appendix 1.

Experimental

The sample were tested using the Revolution Powder Analyzer, which utilizes a rotating drum with transparent sides to measure the flowability static charging of powders. During the tests, a powder volume of 25 cm^3 or 100 cm^3 was loaded into the drum. As the drum was rotated on a pair of motor-driven rollers at pre-defined rotation rates, the avalanche profile of the powders was captured using a digital camera, with the assistance of cold-cathode back-light illumination, as shown in Figure 1. The images of the avalanche profile were collected and processed at 20 frames per second to capture the exact motion of the powder sample. From the images collected, image analysis was then conducted, and numerous parameters were measured or calculated. A reference mask allows for calibration of the imaging system. Several testing modes are available on the Revolution Powder Analyzer, which include flow, packing, and multi-flow and static charging tests [11]. Each of the tests examines a unique aspect of powder properties. The powders in this case were tested with the flow and static charge tests.

Figure 1. Illustration of working principle of Revolution Powder Analyzer. Avalanche profile of powders is captured by a camera as the drum is rotating. The avalanche angle α is measured by image analysis

In the flow test, a relatively low rotation rate 0.3 rotations per minute (rpm) is used, and the details on the avalanche behaviors are captured. The instrument captures 100 avalanches and provides averaged results. Flow properties of the powder can be evaluated by measuring the avalanche angles, which are captured by the camera when the powder avalanche begins. The break energy represents the maximum energy level of the powder test portion before an avalanche begins. The avalanche energy is the amount of energy released during an avalanche. The dynamic density can be calculated from the measured mass of the powder and the bulk volume determined using image analysis tools as the drum is rotating. The cohesion-T is the average of the shear stress overcome by the flowing layer as the powder moves during avalanche.

A low drum speed for the testing has been used for two reasons. One reason is that the speed of the particles in the powder bed in the spreading area of typical printers has been observed to be low for many printers. The other reason is that low speed tests are more sensitive to small changes in the test powder. This is derived from 20 years of testing with the instrument. Low speed tests emphasize particle to particle interactions in the powder as opposed to higher speeds which are more about the dilation of the powder bed due to higher particle velocity and aeration.

Using the ION Charge Module with the Revolution allows the measurement of charge acquisition properties between contact surfaces and test samples while controlling velocity and contact time. The Revolution Powder Analyzer uses a rotating drum with various sides to measure the tribocharging properties of powders. The operator begins the charge test by filling the rotating drum with 100 cc of powder. A motor rotates two high precision rollers that in turn rotate the drum. A charge sensor [Figure 2] measures the charge on the drum side before rotation begins, during drum rotation for a preset time at a preset speed, and after rotation stops for a preset time.

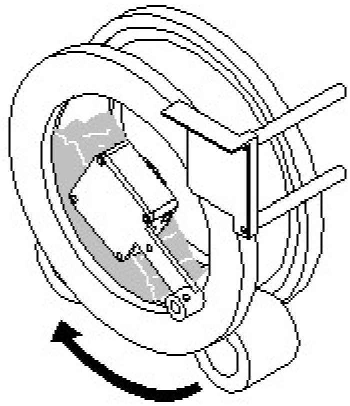

Figure 2. Charge sensor in front of the sample drum

Each sample was tested as is out of the sample container. Samples were overfilled into a 100 cm^3 sample cup then transferred to the test drum. After initial testing the sample was placed in a humid oven at 50 degrees Celsius and 40% relative humidity for 2 hours. Samples were mixed every 30 minutes to expose more powder to the oven humidity. The sample was then retested. The sample was then placed in a 100 degrees Celsius dry oven for 12 hours and a 200C dry oven for 2 hours with mixing every 30 minutes. The sample was cooled to room temperature then retested.

25 cm^3 of the as is sample was tested in a smaller sample drum. The remaining 75 cm^3 of sample was poured into a closed core flow funnel. A core flow funnel has a shallow angle so that the powder flows in a first in last out pattern. The funnel was then opened and the powder flowed into a second closed core flow funnel. The second funnel was then opened and the powder flowed into a third closed core flow funnel. The final funnel was then opened and the first 25 cm^3 of powder exiting the funnel was collected and tested. This procedure was repeated with the dry sample.

The dry samples were recombined and 100 cm^3 of each sample was transferred to a flat pan. The pan was shaken from side to side for 60 seconds to create motion in the sample. The top layer of the sample was then scraped from the powder bed and collected. Then the lower layer was collected. 25 cm^3 of each layer was then tested.

Results and Discussion

A summary of the flow measurements made on the individual samples is given in Table 1. These various measurements were highlighted by the avalanche energy, avalanche angle, and cohesion-T values describing the shear properties of the powder layers during the avalanche were extracted. Generally, lower values for each of these metrics are considered indicative of better powder flow properties [2,3]. The dynamic density and volume fraction of the powder, which captured the packing density of the powder as the drum was rotated, was also measured. Unlike the other properties, an increase in dynamic density can be correlated with better flow properties.

Table 1. Summary of the powder flow test data for each sample

Sample	Avalanche Energy mJ/kg	Avalanche. Angle deg	Cohesion-T Pa	Dynamic Density g/ cm^3	Volume Fraction
Set A Virgin As Is	9.8	30.5	29.0	4.60	0.574
Set A Virgin Humid	9.1	30.2	27.6	4.60	0.574
Set A Virgin Dry	9.9	31.6	30.0	4.67	0.583
Set A Recycled As Is	17.7	36.5	90.6	4.23	0.529
Set A Recycled Humid	19.7	46.3	97.0	4.23	0.528
Set A Recycled Dry	13.0	34.3	35.3	4.38	0.547
Set B Recycled As Is	16.2	44.3	83.7	4.24	0.529
Set B Recycled Humid	17.7	37.6	81.3	4.25	0.530
Set B Recycled Dry	14.5	36.1	35.3	4.18	0.522
Set B Virgin As Is	28.3	58.0	151.0	4.09	0.508
Set B Virgin Humid	28.0	52.8	165.2	3.98	0.497
Set B Virgin Dry	13.3	41.7	60.2	4.04	0.505

The flow measurements for the virgin and recycled powders in Table 1 captured several key relationships and differences between the different powder conditions. Sample Set A Virgin had the best flow properties of the samples tested with the lowest avalanche energy, avalanche angle, and cohesion-t across all tests. Sample A virgin also has the highest density across all tests. Sample B Virgin has the poorest flow properties and the lowest density across all tests.

The changes in the flow properties between the dried samples and the humidity exposed samples are presented in Table 2. In all samples except sample Set A Virgin, exposure to humidity worsened the flow properties of the powders and drying improved the flow properties of the powders. Sample Set B Virgin had the greatest change in flow properties between the humidity exposed and dried sample followed by sample Set A Recycled. In the case of Set A Virgin humidity exposure and drying caused little change in the flow properties of the sample. This data indicates that humidity exposure will generally worsen the flow properties of these powders but drying will reverse these changes.

For Sample Set B, the virgin material has poorer flowability than the recycled material and is more sensitive to humidity exposure and drying. This means that the virgin material may show more variability in testing and performance depending on its environmental exposure. This could cause the virgin material to be accepted for production in dry conditions but rejected in humid conditions. The virgin material may also perform differently in the printer depending on how dry it is. Once processed and recycled the material displays improved flowability and reduced sensitivity to drying or humidity exposure. Both of these samples were acceptable for printing which means sample B Recycled has a larger window where its flowability will be acceptable.

Table 2. Changes in flow properties between dried and humidity exposed samples

Sample	Avalanche Energy mJ/kg	Avalanche. Angle deg	Cohesion-T Pa	Dynamic Density g/cm^3	Volume Fraction
Set A Virgin	-0.8	-1.4	-2.4	-0.07	-0.01
Set A Recycle	+6.7	+12.0	+61.7	-0.15	-0.02
Set B Recycle	+3.1	+1.5	+46.0	+0.07	-0.01
Set B Virgin	+14.6	+11.1	+105.0	-0.07	0.01

For Sample Set A, the recycled material has poorer flowability than the virgin material and is more sensitive to humidity exposure and drying. This means that the recycled material may show more variability in testing and performance depending on its environmental exposure. This could cause the recycled material to be accepted for production in dry conditions but rejected in humid conditions. The recycled material may also perform differently in the printer depending on how dry it is. The virgin material in this case showed almost no sensitivity to humidity exposure or drying.

The ranking of the flow properties of the humidity exposed samples is presented in Table 3. Sample Set A Virgin has the best flow properties followed by Set B Recycled, Set A Recycled, and Set B Virgin. The ranking of the flow properties of the dried samples is presented in Table 4. The ranking for the dried samples was similar to the ranking for the humidity exposed samples but with Set A Recycled and Set B Recycled switching positions. Sample Set A Virgin had the best flow properties for both dried and humidity exposed samples and Set B Virgin had the poorest flow properties.

Table 3. Ranking of flow properties of humidity exposed samples

Sample	Avalanche Energy mJ/kg	Avalanche. Angle deg	Cohesion-T Pa	Dynamic Density g/ cm^3	Volume Fraction
Set A Virgin	9.9	31.6	30.0	4.67	0.583
Set B Recycled	17.7	37.6	81.3	4.25	0.530
Set A Recycled	19.7	46.3	97.0	4.23	0.528
Set B Virgin	28.0	52.8	165.2	3.98	0.497

Table 4. Ranking of flow properties of dried samples

Sample	Avalanche Energy mJ/kg	Avalanche. Angle deg	Cohesion-T Pa	Dynamic Density g/ cm^3	Volume Fraction
Set A Virgin	9.9	31.6	30.0	4.67	0.583
Set A Recycled	13.0	34.3	35.3	4.38	0.547
Set B Recycled	14.5	36.1	35.3	4.18	0.522
Set B Virgin	13.3	41.7	60.2	4.05	0.505

A summary of the static charging measurements made on the individual samples is given in Table 5. For all of the samples, exposure to humidity caused the charging properties of the powders to move toward the positive direction while drying caused the powders to move toward the negative direction. Anecdotal evidence generally suggests that negative charges tend to cause powders to adhere more to equipment. Sample Set A Virgin displayed bigger changes between humidity exposed and dried samples than Set A Recycled while Set B Recycled displayed bigger changes between humidity exposed and dried samples than Set B Virgin.

Table 5. Summary of the static charging data for each sample

Sample	Glass average V	Glass maximum V
Set A Virgin As IS	356	380
Seta A Virgin Humid	423	462
Set A Virgin Dry	-60	-134
Set A Recycled As Is	58	78
Set A Recycled Humid	69	139
Set A Recycled Dry	-40	-155
Set B Virgin As IS	28	67
Set B Virgin Humid	60	89
Set B Virgin Dry	-60	-134
B Recycled As IS	121	149
B Recycled Humid	167	283
B Recycled Dry	-38	-226

A summary of the funnel segregation test data made on the individual samples is given in Table 6. A summary of the changes in the flow properties before and after funnel segregation stress is given in Table 7. None of the samples changed a great deal when exposed to segregation stress using multiple core flow funnels. Sample Set B Virgin displayed the most change and Set A Recycled show more change than Set A Virgin. The dried samples displayed more change than the As Is samples.

Table 6. Summary of the Funnel Segregation data for each sample

Sample	Avalanche Energy mJ/kg	Avalanche Angle deg	Cohesion-T Pa	Dynamic Density g/cm^3	Volume Fraction
Set A Virgin As Is	6.0	31.6	15.09	4.76	0.595
Set A Virgin Segregated	6.6	31.9	16.2	4.76	0.594
Change	+0.6	+0.3	+1.1	-0.00	-0.001
Set A Virgin Dry	6.1	32.7	15.6	4.84	0.598
Set A Virgin Dry Segregated	7.7	32.5	18.7	4.69	0.584
Change	+1.6	-0.2	+3.1	-0.15	-0.014
Set A Recycled As Is	9.7	39.1	50.3	4.37	0.544
Set A Recycled Segregated	10.2	40.1	53.4	4.37	0.545
Change	+0.6	+1.0	+3.0	+0.01	+0.001
Set A Recycled Dry	7.1	36.7	19.6	4.20	0.541
Set A Recycle Dry Segr.	7.6	35.4	33.8	4.52	0.565
Change	+0.5	-0.2	+14.2	+0.32	+0.024
Set B Virgin As Is	13.3	45.9	81.6	4.22	0.527
Set B Virgin Segregated	14.5	47.5	89.8	4.20	0.525
Change	+1.2	+1.6	+8.2	-0.08	-0.002
Set B Virgin Dry	6.3	33.0	32.5	4.20	0.524
Set B Virgin Dry Seg	12.0	41.8	64.0	4.00	0.499
Change	+5.7	+8.8	+31.5	-0.20	-0.025
Set B Recycled As Is	10.0	39.5	42.9	4.43	0.553
Set B Recycled Segregated	10.4	39.5	43.5	4.46	0.557
Change	+0.4	+0.0	+0.5	+0.03	+0.004
Set B Recycled Dry	9.0	32.2	18.1	4.37	0.556
Set B Recycled Dry Segr.	8.3	36.1	30.5	4.31	0.538
Change	+0.5	-0.2	+14.2	+0.06	+0.024

Table 7. Summary of the changes before and after funnel segregation stress

Sample	Avalanche Energy mJ/kg	Avalanche Angle deg	Cohesion-T Pa	Dynamic Density g/cm^3	Volume Fraction
Set A Virgin As Is	+0.6	+0.3	+1.1	-0.00	-0.001
Set A Virgin Dry	+0.5	-0.1	+0.5	+0.04	+0.005
Set A Recycled As Is	+0.6	+1.0	+3.0	+0.01	+0.001
Set A Recycled Dry	+0.8	-0.8	-5.3	+0.12	+0.016
Set B Virgin As Is	+1.2	+1.6	+8.2	-0.08	-0.002
Set B Virgin Dry	+5.7	+8.8	+31.5	-0.20	-0.025
Set B Recycled As Is	+0.4	+0.0	+0.5	+0.03	+0.004
Set B Recycled Dry	+0.5	-0.2	+14.2	+0.06	+0.024

Table 8. Summary of the Pan Segregation data for each sample

Sample	Avalanche Energy mJ/kg	Avalanche Angle deg	Cohesion-T Pa	Dynamic Density g/cm^3	Volume Fraction
Set A Virgin Dry Seg Top	6.7	32.8	18.7	4.64	0.580
Set A Virgin Dry Seg Bottom	7.2	32.7	19.1	4.68	0.585
change	-0.5	0.1	0.4	-0.04	+0.005
Set A Recycled Dry Seg Top	8.6	36.9	40.1	4.40	0.550
Set A Recycled Dry Seg Bottom	7.8	36.1	34.8	4.27	0.534
change	+0.8	-0.8	-5.3	+0.13	+0.016
Set B Virgin Dry Seg Top	14.9	47.4	80.7	4.09	0.511
Set B Virgin Dry Seg Bottom	14.7	47.4	80.6	4.09	0.511
change	-0.2	-0.0	-0.1	0.00	+0.00
Set B Recycled Dry Seg Top	6.6	36.5	31.1	4.27	0.533
Set B Recycled Dry Seg Bottom	7.7	36.5	37.8	4.30	0.537
change	+1.1	0.0	+6.7	+0.03	+0.004

A summary of the pan segregation test data made on the individual samples is given in Table 8. None of the sample changed a great deal when exposed to segregation stress using the pan method. However larger particles were visible on the top layer of all of the samples except for Set B Virgin. This can be seen in Figure 4 for Set B Recycled. Sample Set A Virgin and Set B Virgin showed no change while Set A Recycled and Set B Recycled showed small changes. The data indicates that the flow of these materials is not very sensitive to small changes in the concentration of the larger particles in the sample. The test sample for the top pan was collected by scraping the top half of the material with a flat knife. Therefore the top samples included significantly more than just the top layer of larger particles.

Figure 4. Image of the segregation pan top surface for Set B Recycle after sifting. Larger particles can be seen on the top surface.

The segregation data indicates a difference in how the material change when exposed to segregation stress. Even though the changes were small, they display that recycled and virgin materials have different sensitivities to segregation. These differences could be amplified or localized in production environments where a small percentage of large or small particles can block spreaders and cause streaks in layers.

Summary and Conclusions

In considering the humidity exposure, drying, and segregation, the test data displays that the tested powders have both sensitivity and varying sensitivity to environmental exposure and handling as they are used. In a small sample, this sensitivity generally will change the entire sample portion. In a production environment the entire sample portion or only a subsection of the portion may be affected. This can explain why some materials work well, some material work well then stop working and some materials create layers of unequal size and quality. Typically when AM parts are cross sectioned the measured melt layer thickness is not the same for every layer.

References

[1] M.Z. Gao, B. Ludwig, T.A. Palmer, Impact ofatomization gas on characteristics of austenitic stainless steel powder feedstocks for additive manufacturing, Powder Technol. In press (2020). https://doi.org/10.1016/j.powtec.2020.12.005.

[2] A.T. Sutton, C.S. Kriewall, S. Karnati, M.C. Leu, J.W. Newkirk, Characterization of AISI 304L stainless steel powder recycled in the laser powder-bed fusion process, Addit. Manuf. 32 (2020) 100981. https://doi.org/10.1016/j.addma.2019.100981.

[3] A.B. Spierings, M. Voegtlin, T. Bauer, K. Wegener, Powder flowability characterisation methodology for powder-bed-based metal additive manufacturing, Prog. Addit. Manuf. 1 (2016) 9–20. https://doi.org/10.1007/s40964-015-0001-4.

[5] Thornton, Particle Size and Shape Evaluation of SS-316L Powders from Interlaboratory Study, IJPM,Vol56, No 4,2020,pp41-57

[6] Saad, Jack G, and Thornton, Tony, Paper 097 presented during Session 22, AMPA 2018 Conference, San Antonio, TX.

[7] Mercury Scientific, Revolution Powder Analyzer User Manual, (2010) 248.

Appendix 1. Summary of the Particle Size data for each sample

Sample	Wet Static Laser Light Scattering D_{10} (µm)	Wet Static Laser Light Scattering D_{50} (µm)	Static Laser Light Scattering D_{90} (µm)
Set A Virgin	28.01	46.83	58.49
Set A Recycled	29.01	47.58	59.92
Set B Virgin	26.49	41.55	58.23
Set B Recycled	24.83	42.02	58.96