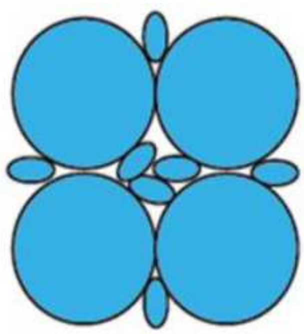
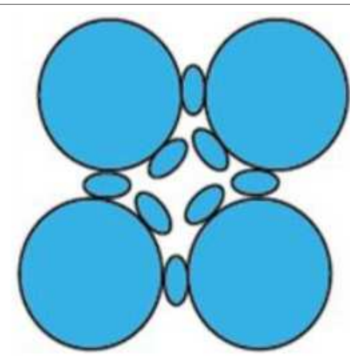
Testing Powders for Additive Manufacturing Applications

The Revolution Powder Analyzer has been used extensively to test the flow properties of metal and polymer powders used for additive manufacturing applications. The tests that have been proven to be suited to additive manufacturing applications include the flowability test, the packing test, the multi-flow test, caking test,and the electrical charge analysis.

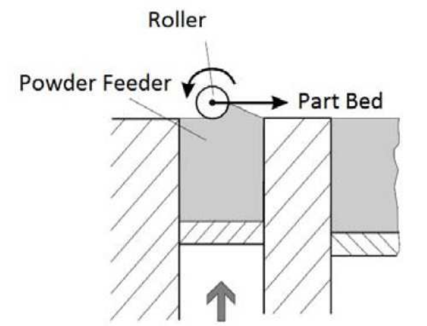
The flow properties of powders are important for AM applications because they determine how the powder will behave in the AM machine. The better and more consistently the powder flows the better it will create layers in the manufacturing process.

	
Good flow means more efficient and homogeneous packing	Poor flow means gaps and in-homogenous packing

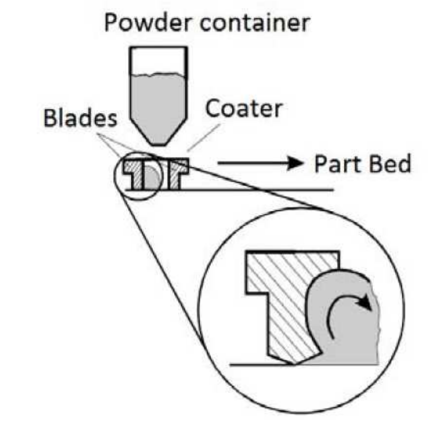
Images from **Characterizing the Bulk & Flow Behaviour of LS Polymer Powders** *Stefan Ziegelmeier-ab, Frank Wöllecke-a, Christopher Tuck-b, Ruth Goodridge-b, Richard Hague-b* *a*BMW Group, Rapid Technologies Center, Germany, Knorrstraße 147, 80788 Munich bUniversity of Nottingham, Mechanical, Materials and Manufacturing Engineering, Nottingham, UK, NG7 2RD

The Revolution Powder Analyzer is uniquely suited to AM applications due to the way the system measures the powders. The powders are tested in a rotating or vibrating sample drum that closely approximates the stress levels on the powder in the printer.

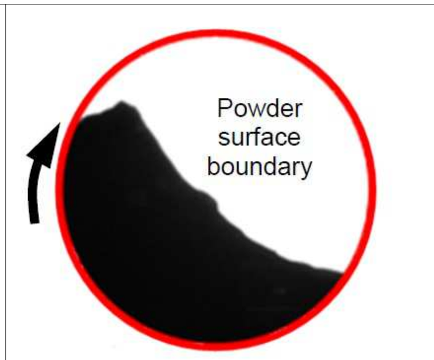
"The boundary conditions generated for the powder surface inside the drum emulate the typical front stress free turning powder wedge behavior generated by any of the actual SLS spreading systems, i.e., the counter-clockwise rotating roller (3DSystems) or the concave blade coater (EOS)"*



Powder Bed System*



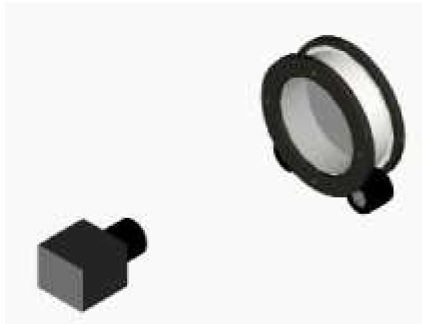
Powder Coating System*



Revolution Test Drum*

*quote and drawing from **ADVANCES IN SLS POWDER CHARACTERIZATION**, A. Amado*, M. Schmid, G. Levy and K. Wegener, Department of Mechanical and Process Engineering, Swiss Institute of Technology, Zurich 8008, Switzerland and Inspire AG, irpd Institute, St. Gallen 9014, Switzerland

The Revolution Powder Analyzer uses a rotating and vibrating drum to measure the flowability potential of powders. The operator begins the tests by filling the rotating drum with from 8 to 100 cc of powder. A motor rotates two high precision rollers that in turn rotate the drum. A digital camera with the assistance of cold cathode back-light illumination takes digital images of the powder during the rotation process. From the images collected, the software measures the behavior of the powder due to the drum rotation and how this behavior changes over time.



Filling the sample drum

Sample Drum on Rollers

The Revolution Powder Analyzer is very easy to use with the operator simply loading the powder into the sample drum, putting the drum in the instrument, and starting the sample test. Tests require roughly 5 to 10 minutes and can be linked together to run multiple tests without operator intervention. For example, for AM powders, users typically run a charge analysis followed by a flow analysis followed by a packing analysis as a linked method.

Revolution Powder Analyzer highlights for AM
The Revolution Powder Analyzer is very easy to understand and the results produced by it are very easy to interpret.
The Revolution Powder Analyzer is the only powder flow tester that can measure the flowing density of the sample powder and the speed dependence of the flowing density.
The Revolution Powder Analyzer is the only powder flow tester that can measure flow, vibrational packing, and electrical charging in one test cycle.
The Revolution Powder Analyzer is the only powder flow tester that can measure the smoothness (surface fractal) of the surface of the powder bed.
The Revolution Powder Analyzer is the only powder flow tester that can measure avalanche angle, avalanche energy, and break energy.
The Revolution Powder Analyzer requires no preventative maintenance and does not require periodic calibration.
The Revolution Powder Analyzer is the only powder flow tester that can heat the sample to 250C with the optional drum heater.

The Flow Test

The Revolution flow test measures how powders flow under low speed and low stress gravitational conditions. The main measurements are avalanche energy, break energy, avalanche angle, dynamic density, and surface fractal. The avalanche angle and break energy represent the powder's resistance to flow. The avalanche energy measures how liquid the powder becomes while flowing. The dynamic density measures the density of the powder as it is flowing. This is a good approximation of the density of the powder in front of the spreader or roller. Researcher's have tied the avalanche angle to powder bed defect rates.

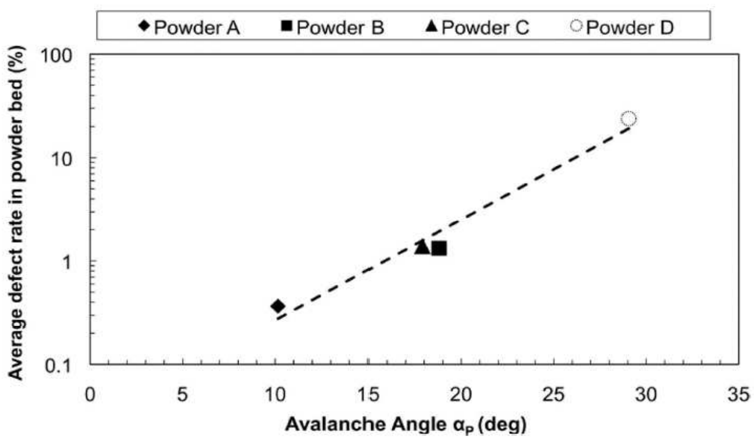


Fig. 15. Effect of powder flowability on powder bed defects rate.

Image from “**Development of Evaluation technique for Powder Bed Quality is SLM Process by Image Processing**” by Shinnosuke Yamada and Nobuyuki Tkahashi.

The surface fractal measures the smoothness of the surface of the powder bed. Some researchers have correlated the surface fractal with the smoothness of the final part.

The flow test is used to compare the flowability of AM powders from different manufacturers, different powder formulas, and different amounts of flow additives. The flow test is also useful for studying the effects of powder re-use and the effects of blending virgin powder with used powder. For polymers, the effects of temperature on powder flow can also be studied with the Revolution's optional drum heater.

Comparing Powders that Failed From Different Manufacturers

Three sets of powders are compared. All of these powders showed a failure in manufacturing parts after some time. Samples of the failed powders were then collected and analyzed.

Powder Supplier A

Sample	Avalanche Energy	Energy Change	Avalanche Angle	Angle Change	Dynamic Density	Density Change
Supplier A Virgin	13.8 mJ/kg		41.7 deg		4.18 g/cc	
Supplier A Fail	45.7 mJ/kg	239.7%	57.1 deg	36.9%	4.10 g/cc	-1.9%

Powder Supplier B

Sample	Avalanche Energy	Energy Change	Avalanche Angle	Angle Change	Dynamic Density	Density Change
Supplier B Virgin	15.1 mJ/kg		44.6 deg		4.21 g/cc	
Supplier B Fail	26.0 mJ/kg	72.3%	50.0 deg	12.1%	4.12 g/cc	-2.2%

Powder Supplier C

Sample	Avalanche Energy	Energy Change	Avalanche Angle	Angle Change	Dynamic Density	Density Change
Supplier C Virgin	15.7 mJ/kg		42.5 deg		4.44 g/cc	
Supplier C Fail	44.9 mJ/kg	185.4%	54.6 deg	28.5%	4.14 g/cc	-6.8%

The virgin powders from these three manufacturers had similar flow properties. Supplier A had the best initial flow properties but not the highest density. Upon use however Sample A had the greatest change in avalanche energy and avalanche angle. Sample B showed the least change on failure. The density of all of the failed samples was in the 4.10-4.14 g/cc range and the avalanche angle was 50 degrees and over.

Measuring Changes In Flow Properties With Use

Virgin and used 316 powder

Sample	Avalanche Energy	Avalanche Angle	Dynamic Density	Surface Fractal
Virgin	6.0 mJ/kg	31.4 deg	4.54 g/cc	2.3
Used 2 Times	15.9 mJ/kg	34.8 deg	4.46 g/cc	2.3
Used 5 Times	20.5 mJ/kg	39.7 deg	4.47 g/cc	2

The sample's main flow properties worsened as the powder was used with the avalanche energy and avalanche angle increasing. The dynamic density decreased from the virgin sample. Clearly the use of the powder is changing its flow properties. In this case the flow change is due to satellite particles sticking to the main particles. This change in powder was not evident in other tests like particle size of hall flow meter.

Virgin and Used Polymer Powder

Sample	Avalanche Energy	Avalanche Angle	Dynamic Density	Surface Fractal
Virgin	11.3 mJ/kg	42.7 deg	0.452 g/cc	2.2
Used 2 Times	16.7 mJ/kg	47.2 deg	0.420 g/cc	3.1

The sample's main flow properties worsened as the powder was used with the avalanche energy and avalanche angle increasing. The dynamic density also decreased with use.

Virgin and Used Ti64 Powder

Sample	Avalanche Energy	Break Energy	Dynamic Density	Surface Fractal
Virgin Powder	20.5 mJ/kg	29.2 mJ/kg	2.57 g/cc	5.3
Used Powder	16.5 mJ/kg	25.6 mJ/kg	2.59 g/cc	3.3

The sample's main flow properties improved as the powder was used with the avalanche energy and break energy decreasing. The dynamic density increased slightly with use. For this application, the process of making parts disproportionately removes smaller particles. This loss of small particles improved the flowability of the sample.

Measuring Changes In Flow Properties With Blending Virgin and Used Material

Virgin and blended inconel

Sample	Avalanche Energy	Break Energy	Dynamic Density	Surface Fractal
Virgin	13.9 mJ/kg	38.7 mJ/kg	4.09 g/cc	4.4
20% Virgin-80% Used	14.7 mJ/kg	39.1 mJ/kg	4.10 g/cc	4.6
50% Virgin-50% Used	18.7 mJ/kg	45.1 mJ/kg	3.84 g/cc	4.2

The virgin sample had the best flow properties with the lowest avalanche energy and break energy. The 20% Virgin 80% Used powder blend had similar flow properties to the virgin material with slightly poorer flowability. The 50% Virgin 50% Used powder blend had much poorer flow properties than the other samples and a lower density.

Powder behavior can be difficult to predict and does not always produce the expected or linear results. In this case the expected result would be that the more virgin material in the blend the closer the blend would be to the virgin material.

Comparing Similar Powders From Different Manufacturers

316 10-45 micron powder from three suppliers

Sample	Avalanche Energy	Avalanche Angle	Dynamic Density	Surface Fractal
Supplier A	4.1 mJ/kg	28.5 deg	4.52 g/cc	1.8
Supplier B	10.3 mJ/kg	35.8 deg	4.54 g/cc	2
Supplier C	17.9 mJ/kg	43.9 deg	4.07 g/cc	3.1

The powder from Supplier A flows better than the powders from the other suppliers and produced a smoother surface. Some manufacturer's are better than others at improving the flowability of their powders. Users should purchase the powder's that have the best flow properties. Powder manufacturers should study the factors that affect the flow of their materials to produce the best flowing powders possible.

Comparing Different Formulas And Lots From The Same Manufacturer

Different lots of 316 10-45 micron powder from the same manufacturer

Sample	Avalanche Energy	Avalanche Angle	Dynamic Density	Surface Fractal
Formula 1	9.2 mJ/kg	34.2 deg	4.65 g/cc	1.8
Formula 2	11.5 mJ/kg	36.5 deg	4.38 g/cc	2.2
Formula 3	32.4 mJ/kg	44.6 deg	4.01 g/cc	5.4

Small changes in how a powder is produced can create large changes in its flow properties. All of the above samples are 316 stainless steel with a size distribution from 10-45 microns from the same manufacturer. Formula 1 and 2 have similar flow properties but Formula 1 flows better and reaches a higher flowing density. Formula 3 has poor flow properties. The flow test is also useful for measuring lot to lot variation for both powder and part producers.

Measuring Changes In Flow Properties With Particle Size

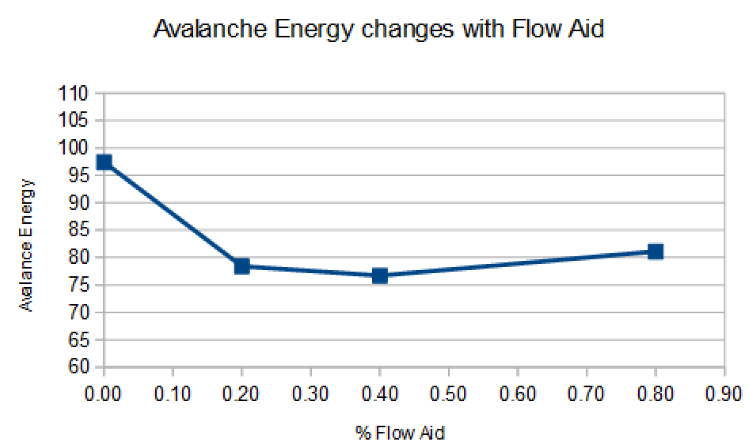
Metal alloy samples

Sample	Avalanche Energy	Break Energy	Dynamic Density	Surface Fractal
40 Micron Mean	12.3 mJ/kg	29.8 mJ/kg	4.21 g/cc	2.3
17 Micron Mean	43.1 mJ/kg	72.7 mJ/kg	3.86 g/cc	4
6 Micron Mean	59.1 mJ/kg	101.1 mJ/kg	3.60 g/cc	6.9

The 40 Micron Mean sample had the best flow properties and the highest density. Generally speaking the smaller the particles the poorer the flow properties and the lower the powder bed density. This is dependent on the size distribution as well as the mean size.

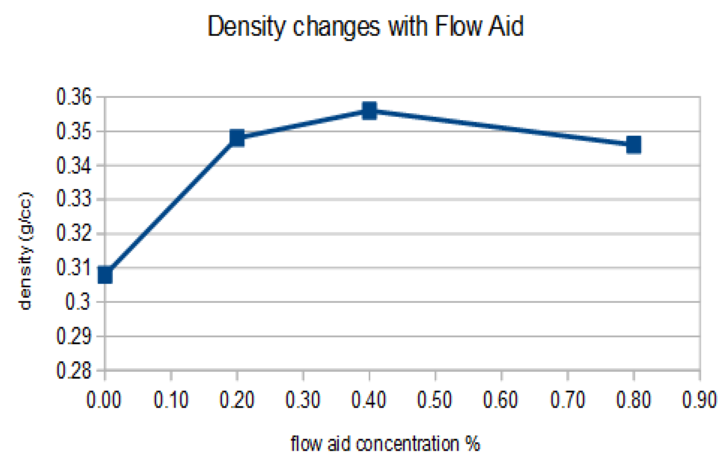
Measuring The Effects Of Additives

Polymer powder with silica flow additive



Polymers used for additive manufacturing usually require a flow additive to make them flow well enough to spread on the printer. Silica is typically used as the flow aid. The challenge is to add the correct amount of flow aid to improve the flow of the material but not enough to affect the strength of the end parts.

For the above sample, the silica flow additive improved the flowability of the powder. This is seen in the lowering of the avalanche energy. The best flow was achieved at 0.4 percent flow additive. The flowability of the powder worsened with additional flow additive. This U-shaped curve is typical for flow additives.



The flow additive increased the flowing density as well as improved the flow of the powder. The maximum density occurred at 0.4 percent flow additive. Adding more additive reduced the density from the maximum.

SS 316 powder with and without additive

Sample	**Avalanche Energy**	**Avalanche Angle**	**Dynamic Density**
Original Material	15.2 mJ/kg	31.9 deg	4.33 g/cc
With Flow Additive	11.5 mJ/kg	35.0 deg	4.53 g/cc

Flow additives are also used for metal powders and show similar behavior to polymers. In the case above the increase in flowability also created an increase in dynamic density due to more efficient packing of the powder particles.

Studying Temperature Effects

The Revolution Powder Analyzer can be equipped with a heated sample drum to test samples at high temperatures.

Avalanche angle of polymer at different temperatures

Sample	26 C	55 C	120 C
Polymer A	46.1 deg	47.5 deg	53.1 deg

Typically polymer powders have poorer flow properties as their temperature increases.

Stainless Steel 316 at different temperatures

Temperature	Avalanche Angle	Dynamic Density	Surface Fractal
26 Celsius original sample	38.1 deg	4.364 g/cc	1.75
250 Celsius	43.2 deg	4.066 g/cc	4.36
26 Celsius after cooling	37.9 deg	4.310 g/cc	2.16

Metal powders can show changes in flowability with temperature even when the temperature is far from the material's melting point. In this case the flowability of the stainless steel sample worsened at 250 degrees Celsius. This can be seen in the increase in Avalanche Angle from 38.1 degrees to 43.2 degrees. In addition, the density decreased with temperature and the surface of the powder became more rough as evidenced by the increased surface fractal. The powder returned close to its original state after cooling.

The Packing Test

The Packing Analysis studies the powder's ability to pack or settle after being exposed vibrational movement. The analysis measures the change in the powder density and the force required to break the powder mass and induce flow after exposure to vibrational forces. The powder is then exposed to high flow speeds to study if the powder returns to its original unpacked density. The packing properties of AM powders have been shown to directly affect the quality of the printed parts.

Different formulas of 316 10-45 micron powder from the same manufacturer

Sample	Volume Reduction	Volume Recovery	Packed Density	Dynamic Density	Packed Angle	Avalanche Angle
Formula 1	-6.60%	99.60%	4.97 g/cc	4.65 g/cc	51.8 deg	34.2 deg
Formula 2	-5.50%	99.30%	4.74 g/cc	4.38 g/cc	47.8 deg	36.5 deg
Formula 3	-2.20%	98.00%	4.08 g/cc	4.01 g/cc	48.4 deg	44.6 deg

In this case, Formula 1 had the most volume reduction and achieved the highest density due to vibration. This is due to the fact that Formula 1 had the best low pressure flow properties as exhibited by the low avalanche angle. Powders that flow well can also pack well under vibration. Formula 3 had the lowest volume reduction and density and also had the poorest flow properties.

Virgin and used Ti64

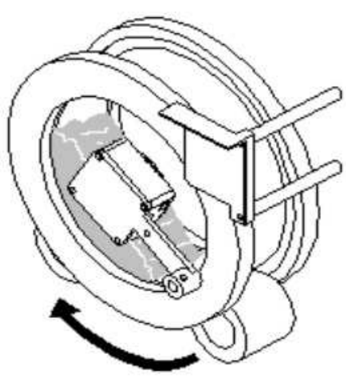
Sample	Volume Reduction	Volume Recovery	Packed Density	Dynamic Density	Packed Angle	Avalanche Angle
Virgin	-9.80%	100.80%	2.74 g/cc	2.46 g/cc	58.5deg	34.6 deg
Used	-10.30%	99.80%	2.87 g/cc	2.54 g/cc	57.6 deg	33.3 deg

For this application, the used powder had more volume reduction and achieved a higher packed density than the virgin sample. This was due to the loss of fine particles as the powder was used. This loss of small particles improved the flowability of the sample allowing it to pack more efficiently.

The Charge Test

Powders and granular materials can acquire electrical charge on the surface of their particles due to contact and movement against handling equipment and containers. They can also acquire charge due to contact and movement of particles within the material itself. This process is called tribocharging. Tribocharging is caused by electrons moving from one surface to another when different materials come in contact with each other. One material will become positive and the other will become negative. The amount of charge developed depends on the nature of the materials in contact, the pressure of the contact, the relative velocity of the contact surfaces, and the friction between the contact surfaces.

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. The charge sensor 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.

	The sample drum is rotated at a programmed velocity and the charge on the contact plate is measured at programmable intervals using a chopper stabilized field meter.

For AM powders, the charge test can be used to study the surface properties of sample powders. Powder charging is sensitive to surface impurities, surface oxidation levels, and additives blended into sample powders.

Comparing Similar Powders From Different Manufacturers

316 10-45 micron powder from 3 different manufacturers

Sample	Average Charge	Max Charge	Charge to Mass
Supplier A	-415 Volts	-469 Volts	-1.0 V/g
Supplier B	-103 Volts	-315 Volts	-0.3 V/g
Supplier C	667 Volts	740 Volts	1.5 V/g

Even with the same base material and same particle size range, powder charging properties can vary widely. This is usually due to differences in the surface properties of the particles making up the powder or to additives in the powder. These surface properties or additives may affect the quality of the printed part.

Comparing Powders With Additives

SS 316 Powder

Sample	Average Charge	Max Charge	Charge to Mass
Original Material	-332 Volts	-377 Volts	-0.8 V/g
With Flow Additive	-33 Volts	-53 Volts	-0.1 V/g

The flow additive reduced the tribocharging potential of the sample powder by a factor of ten. Additives may enhance the flow and charging properties of the materials but their presence may affect the strength of the printed part.

Comparing Virgin and Used Powders

SS 316 powder

Sample	**Average Charge**	**Max Charge**	**Charge to Mass**
Virgin Material	-252 Volts	-293 Volts	-0.63 V/g
Used Powder	-113Volts	-204 Volts	-0.24 V/g

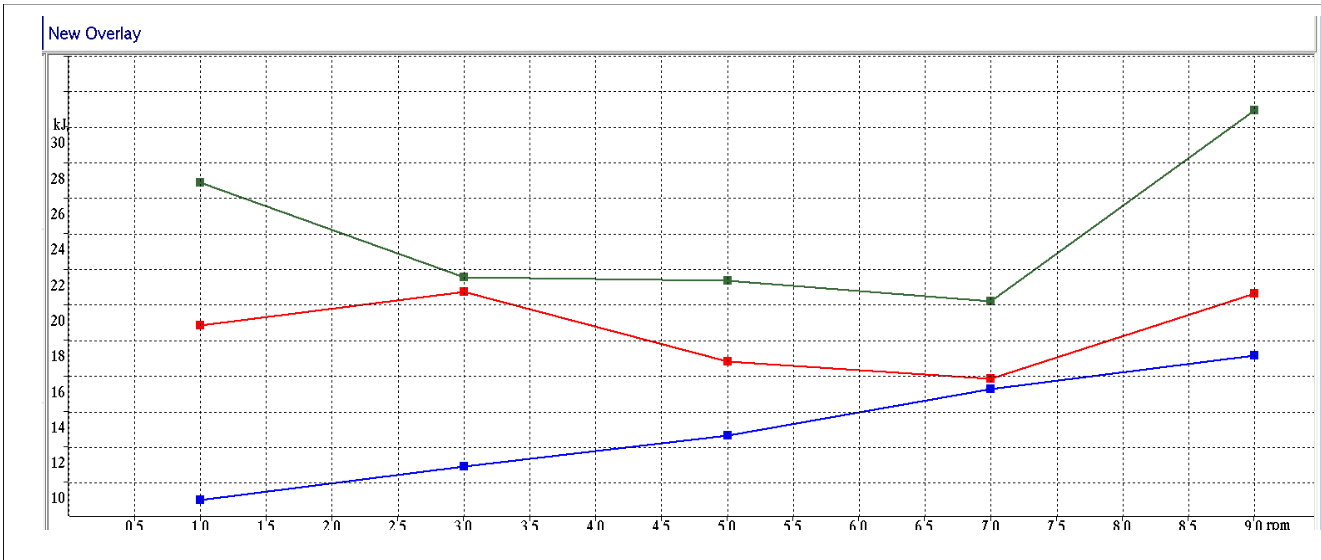
The flow additive reduced the tribocharging potential of the sample powder by a factor of ten. Additives may enhance the flow and charging properties of the materials but their presence may affect the strength of the printed part.

The Multi-Flow Test

Powders can behave very differently depending on the amount of energy they are subjected to as they move through handling equipment. One powder may flow more evenly as it is subjected to more mechanical energy while another powder may become erratic. This behavior can be studied using the Revolution Multi-Flow Test Method. In the multi-flow method, the sample drum speed is increased gradually over time and the sample powder's behavior is measured.

The Multi-Flow Analysis studies how a powder or granular material transitions from avalanching to continually flowing as it is subjected to faster speeds. By gradually increasing the rotation speed in the Multi-Flow Analysis, the user can evaluate the speed at which their powder is no longer avalanching in their process but flowing continuously. This data can be used to predict how powders will behave as the spreading speed increases.

Different formulas of 316 10-45 micron powder from the different manufacturers



Avalanche Energy versus Rotation Speed

Sample	Av Energy Average	Av Energy Slope	Break Energy Average	Break Energy Slope
Supplier A	13.0 mJ/kg	1.0 mJ/kg rpm	28.5 mJ/kg	0.88 mJ/kg rpm
Supplier B	18.6 mJ/kg	-0.10 mJ/kg rpm	38.6 mJ/kg	0.02 mJ/kg rpm
Supplier C	24.2 mJ/kg	0.34 mJ/kg rpm	40.0 mJ/kg	-0.63 mJ/kg rpm

In this case, the powder from Supplier A had lower avalanche and break energies but these went up as the rotation speed increased. The powder from Supplier B was the most stable with flow speed. The powder from Supplier C showed improvement with flow speed up to 7 rpm but had an increase in avalanche energy after that.

The Caking Test

The caking test is used to simulate storage conditions and their effect on sample powders. Powder is tested initially and then it is put in a compression cell and has pressure applied to it for a set time and under various environmental conditions. The pressure used is typically the pressure in the actual storage or shipping container. The environmental conditions typically represent the conditions the sample will be exposed to during shipping and use. After the compression time the sample is transferred to the Revolution sample drum and is retested.

Polymer powder under pressure

Sample	Av Energy Average	Avalanche Angle	Dynamic Density
Fresh	46.1 mJ/kg	56.6 deg	0.516 g/cc
17 hours Ambient	48.2 mJ/kg	56.6 deg	0.533 g/cc
17 hours 40C	49.2 mJ/kg	62.1 deg	0.572 g/cc

The test data indicates that the sample is changing flow properties and density under storage conditions. This is due to agglomeration of the powder particles in the sample. This change is more pronounced as the temperature increases. Temperatures of 40 degrees Celsius are easily achievable during shipping is warm climates and during summer months.

Terms

Avalanche Energy (mJ or pascal or mJ/kg): The avalanche energy is the amount of energy released by an avalanche in the sample powder. It is calculated by subtracting the energy level of the powder after an avalanche from the energy level before the avalanche. The reported avalanche energy is the average avalanche energy for all of the powder avalanches.

Break Energy (mJ or pascal or mJ/kg): The break energy is the maximum energy level of the sample powder before an avalanche begins. This energy level or force represents the amount of energy/force required to start each avalanche. The reported break energy is the average break energy for all of the powder avalanches.

Avalanche Versus Break Energy - The Avalanche Energy is the amount of energy released by an avalanche in the sample powder as the sample drum turns. The Break Energy is the maximum energy level of the sample powder before an avalanche begins. The Break Energy represents the amount of force required to break the sample powder's surface and cause the powder to flow. The Avalanche Energy represents the amount of powder that flows once the powder starts moving. In understanding the break energy and avalanche energy, it is useful to compare powder flow to liquid flow. If a viscous liquid where put in the sample drum and the drum was rotated, the liquid would move to a certain height in the drum and continuously flow. It's break energy would be a constant value depending on the liquid's viscosity. The avalanche energy of the liquid would be zero because it would not avalanche but flow continuously. Therefore the break energy measures resistance to flow for powders similar to a viscosity for liquids. The avalanche energy measures the balance between solid-like and liquid-like behavior in the powder. The closer the avalanche energy is to zero the more liquid-like the powder is behaving and the higher the avalanche energy is the more the powder is behaving as a solid.

Avalanche Angle (degrees): The software collects the angle of the powder at the maximum energy prior to the start of the powder avalanche occurrence. This measurement is the average value for all the avalanche angles. In our avalanche cycle example displayed above, the avalanche angle would be calculated at the peak cycle. The RPA software calculates the flowability angle from the center point on the powder edge to the top of the powder edge. The avalanche angle is not the angle of repose which is the angle when the powder stops flowing.

Rest Angle (degrees): The software collects the angle of the powder at the minimum energy of the powder at the end of the avalanche occurrence. This measurement is the average value for of all the rest angles. The rest angle is typically close to the angle of repose of the powder.

Dynamic Density (g/cc): For every digital image taken, the software measures the density of the sample. The software then calculates the average density of the powder during the test.

Surface Fractal: The surface fractal is the fractal dimension of the surface of the powder and provides an indication of how rough the powder surface is. The measurement is made after each avalanche to determine how the powder reorganizes itself. The standard fractal calculation is used and results are normalized to give a range of 1 to 11. If the powder forms a smooth even surface, the surface fractal will be near two. If the surface is rough and jagged, the surface fractal will be greater than five. For applications requiring an even distribution of powders, such as die filling, the closer the surface fractal is to two the better the powder will perform.