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Flowability of SLS powders

- a frequently neglected parameter -

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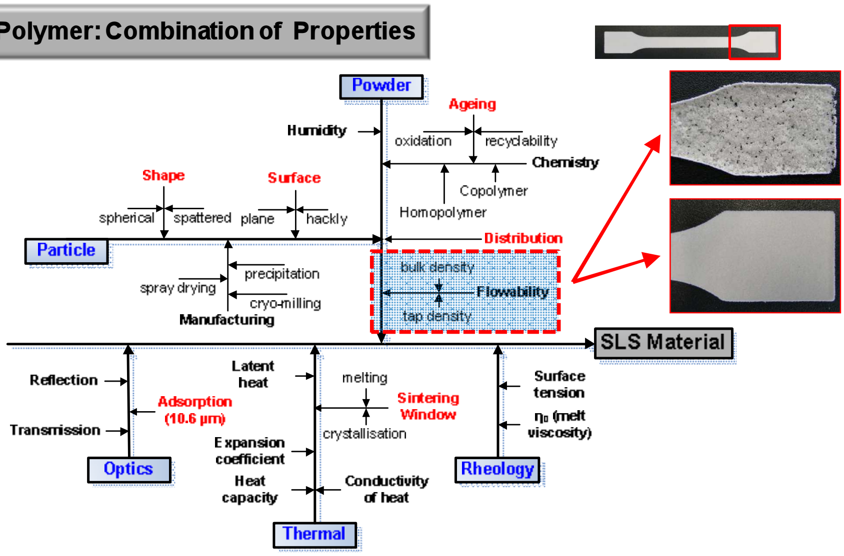
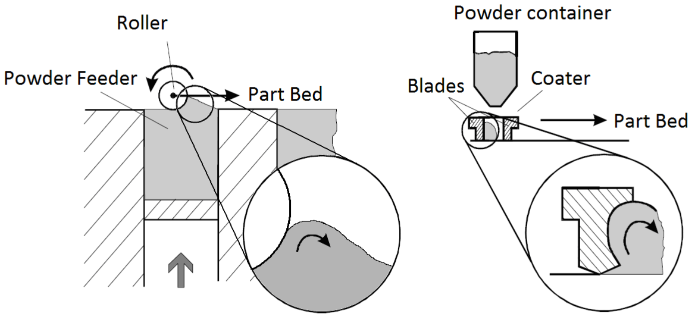
SLS Polymer: Combination of Properties



Powder recoating: Commercial Systems

SLS powder spreading systems: Roller (3DSystems) & Blade (EOS)



Is understanding of flowability of SLS powder a necessary topic?

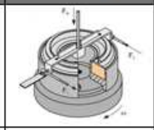
SLS Polymer Powder Development

Production Technology	Intrinsic characterization test				Non-intrinsic characterization test				Author/ Research Group
	DSC	TG	MFI/ Rheo.	*Others	PSD	Particle Shape	Tap/Bulk Density	**Others	
Powder mixing (polymer, fiber, beads)	✓	✗	✗	✓	✗	✗	✗	✗	[7][10][12]
	✗	✗	✗	✓	✗	✗	✗	✗	[13][14][15][16]
	✓	✗	✓	✓	✗	✗	✗	✗	[17]
Melt mixing & cryogenic grinding/spray drying	✓	✗	✓	✓	✓	✗	✗	✗	[18]
	✓	✗	✓	✓	✗	✗	✗	✗	[19]
	✓	✓	✓	✓	✓	✗	✗	✗	[11][20]
Dissolution-precipitation	✓	✓	✗	✓	✓	✗	✗	✗	[21]
	✓	✓	✓	✓	✓	✗	✗	✗	[22]
	✓	✓	✓	✓	✓	✗	✗	✗	[23]
Mechano-chemical alloying/ Solid state	✗	✗	✗	✓	✓	✗	✓	✓	[6][8]
	✓	✗	✓	✓	✓	✗	✓	✗	[9]
	✓	✗	✗	✓	✓	✗	✗	✗	[24]

Other characterization methods: *FTIR, EDX, XRD, etc.; **Angle Of Repose, Carr Index, etc.

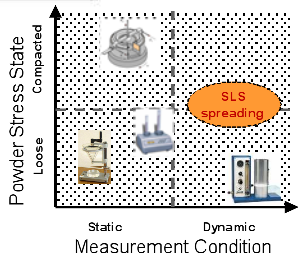
- In general, extrinsic properties are barely reported or just not considered
- Trial & error tests dominate over any preliminary characterization
- Several authors achieve adequate intrinsic properties for processing, but fail during the spreading step

Measurement of powder behaviour

Method	Bed Expansion Ratio	Angle of Repose	Ring Shear Cell	Bulk/Tap Density
				
Measurement Condition	Dynamic under vertical fluid drag load	Static under free external load	Quasi-Static under pressure	Static under the effect of powder weight
Characterization parameters	Fluidized height v/s upstream fluid flow	Pile angle	Shear force v/s normal pressure/ compression rate	Loose and packed height v/s n° of taps
Standard	Not standardized	DIN ISO 4324	ASTM D6773	ASTM D7481

Which system suites best for powder characterization towards SLS?

- No general theory for powder behaviour is available
- Results strongly dependent upon the powder stress condition and packing (H_R = Bulk/Tap widely used)
- Systems present complementary information
- More accurate results for systems that emulate the final handling condition

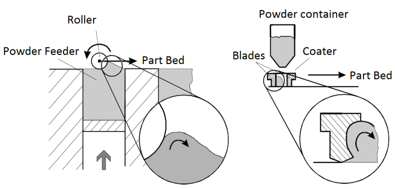


Experimental Device & Methodology

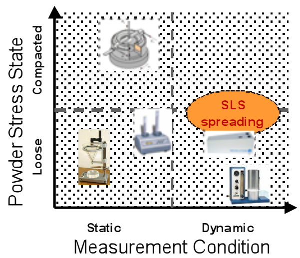
© Revolution Powder Analyzer (RPA)
Mercury Scientific Inc

Schematic diagram of rotating and image acquisition system

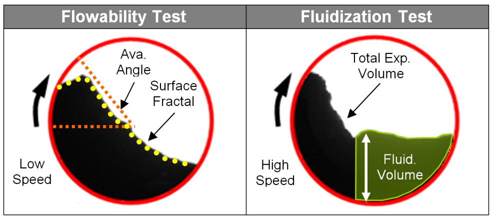
- Similar dynamic handling
- Near stress state condition
- Allows adjustments at different processing speeds

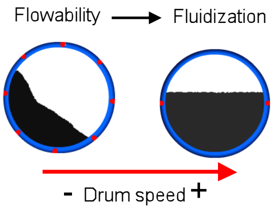


SLS powder spreading systems: Roller (3DSystems) & Blade (EOS) (adapted from [28])



Experimental Device & Indexes





Test	Index	Definition
Flowability	Avalanche Angle	Angle obtained from a linear regression of the free surface at the maximum potential energy prior to the start of the powder avalanche occurrence
	Surface Fractal	Fractal dimension D obtained from the free surface of the powder. D corresponds to a dimensionless parameter based on the self-similarity concept and constitutes a powder rearrangement indicator
Fluidization	Total Volume Expansion Ratio	Ratio between the total volume measured inside the drum (expanded volume) and the volume occupied by the powder in the preparation sample container (tap density volume: 25 cc)
	Fluidized Volume	Fraction of the total volume that develops a fluidized state defined by quasi-horizontal powder surface inside the drum

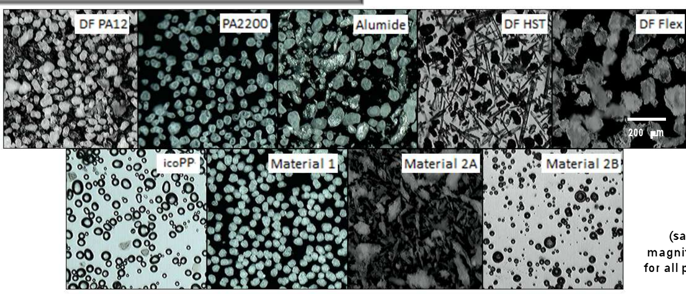
Surface Fractal D: $L(\varepsilon) = M\,\varepsilon^{(1-D)}$

L : length estimate
ε : measurement scale

Experimental Device & Methodology

Commercial Materials

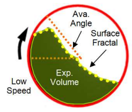
Research Materials



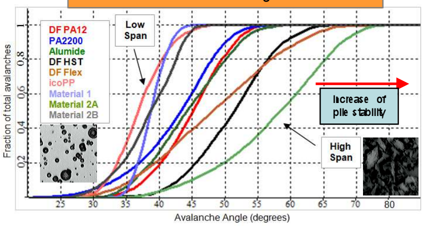
(same magnification for all pictures)

Material	DF PA12	PA 2200	Alumide	DF HST	DF Flex	icoPP	Material 1	Material 2A	Material 2B
Base Material	PA12	PA12	PA 12	PA12	TPE*	coPP	PA	PA	PA
Filler	None	None	Aluminium	Mineral Fibers	N/A	None	None	Nano-Silica	Nano Silica
Compound Method	Neat Polymer	Neat Polymer	Mech. Mixing	Mech. Mixing	Neat Material	Neat Polymer	Neat Polymer	Mechano Fusion	Spray Drying
Final Compound	1 Phase	1 Phase	2 Phases	2 Phases	1 Phase	1 Phase	1 Phase	1 Phase	1 Phase
PSD (µm)	D10=44 D50=56 D90=76	D10=39 D50=53 D90=72	D10=40 D50=65 D90=85	Not defined**	D10=64 D50=88 D90=98	D10=36 D50=58 D90=88	D10=40 D50=48 D90=65	D10=45 D50=97 D90=115	D10=22 D50=44 D90=70

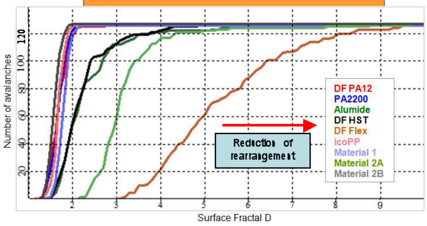
Results & Analysis: Flowability



Cumulative Avalanche Angle Distribution



Cumulative Surface Fractal Distribution

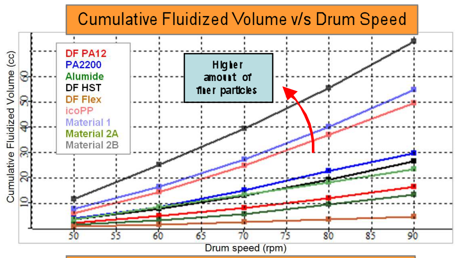


- Materials with a near spherical shape & 1 phase present the lowest avalanche angle and a narrow distribution (span).
- Powders with a broader particle morphological distribution increase their average value
- A second phase (mixture) has a mayor influence when different particle aspect ratios are present (HST v/s Alumide)
- Highly geometrical distorted particles depict a higher d_{50} and span

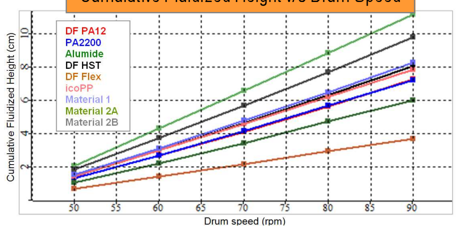
- Materials with near spherical and broader distribution of shapes present the lowest surface fractal value (better rearrangement after an avalanche)
- Compounds with 2 heterogeneous phases present similar and higher surface fractal results (HST & Alumide). No differences regarding aspect ratio can be appreciated.
- In comparison to previous results, DF Flex presents even a worse powder rearrangement than Material 2A

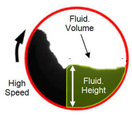
Results & Analysis: Fluidization

Cumulative Fluidized Volume v/s Drum Speed



Cumulative Fluidized Height v/s Drum Speed





- Both variables (fluidized volume and height) present a linear correlation regarding drum speed
- Powders with finer particles present a higher volume slope
- Similar trend can be observed for the fluidized height
- DF Flex presents the lowest degree of fluidization

SLS-Materialien: Summary

- A new powder characterization system has been introduced under near SLS spreading stress state when the powder is mechanically agitated inside a rotational drum (system called Revolution Powder Analyzer was employed).
- New aspects regarding the dynamic powder behavior were analyzed.
- Different commercial powders and others under current research were studied.
- A new aspect regarding powder rearrangement was coupled to a traditional measurement index to improve the description of the powder flowing performance.
- This research was limited to new materials. Properties after recycling and the behavior at the chamber processing temperature are going to be address in a further study.
- These results can be used to complement the existing methods to achieve a more accurate understanding about SLS powder suitability and thus reduce the powder development cycle time.