Non-Standard Powder Rheology with Standard Rheometer Equipment

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ABSTRACT

Powders are used in many different industries and their flow behaviour is an important property during production processes and for the final product. The final product does not have to be a powder itself. In the manufacture of many products, additives or ingredients are added in powder form. Spices are added to food, carbon black is added to rubber and so on. In all these cases, powders must be stored, transported, and dosed during the manufacture of the various products. When planning the equipment for storing and handling of powders, their flow behaviour and compactability are required.

Dedicated instruments with special measuring geometries are available for the characterization of powders. But it is also possible to use a modern rheometer for this purpose, as long as it has a normal force sensor and can run stress-controlled shear measurements.

MATERIALS AND METHODS

The powders used in this study were two different stearates, substances used e.g., as additives for lubricants or release agents. The aim of the tests carried out was to quantify the compacting behaviour and to compare the onset of powder flow of the two samples tested.

All tests were performed with a Thermo Scientific™ HAAKE™ MARS™ 60 Rheometer. The rheometer was equipped with a Peltier temperature module for cone and plate- or parallel-plates geometries. For the measurements on powders, a serrated parallel plates geometry has been used to avoid slippage on the sample surface. For simplicity, the same geometry has also been used for the compression tests although standard parallel plates would have been sufficient here. Parallel-plates geometries should always be used for compression tests, to exert a pressure as uniform as possible across the sample area.

A shallow cup ring was placed onto the lower geometry plate to create a cylindrical sample compartment with a depth of 2.3 mm[1]. A powder was poured into the compartment and the excess powder was pulled off with a straight edge (**Fig. 1**). The use of this loading method achieved samples with 2 parallel surfaces and uniform densities.

FIGURE 1: Sample preparation with the shallow cup ring

For the compression tests, the upper plate was brought to a position 1 mm above the sample surface and afterwards moved downwards with an axial speed of 0.1 mm/s until an axial force of 50 N was reached.

To quantify the tendency of the powders to flow, amplitude sweeps, and stress ramps have been used, methods similar to the determination of the yield stress.

For amplitude sweeps, the samples were consolidated at 20 °C with 5 minutes of compression at an axial force of 5 N, which corresponds to a normal stress of 15.9 kPa. Afterward, an amplitude sweep was performed in Controlled Deformation (CD) mode in a range from 0.001 % to 100 %. During the test, the axial force was kept constant at 5 N.

The same sample consolidation procedure was performed before running the stress ramps. The stress was increased on a logarithmic base from 1 Pa to 10.000 Pa in 5 min, again maintaining the axial force of 5 N throughout the measurement. The basic concept of this method is also used in other test methods to characterize the bulk properties of powders e.g., ASTM D7891[2]. However, since the measuring geometries and the test parameters are different, all these methods will give different values.

Depending on the individual application, the applied axial force during sample consolidation and testing can be adjusted to mimic real conditions as closely as possible.

RESULTS

The results of the compression tests showed very good reproducibility, confirming that the shallow cup ring is a helpful tool to prepare powder samples in a reproducible way. Since the compression tests focus on the sample height and not on the mechanical strength of the powders, like the amplitude sweeps and the stress ramps do, the term compression is used here instead of consolidation[3].

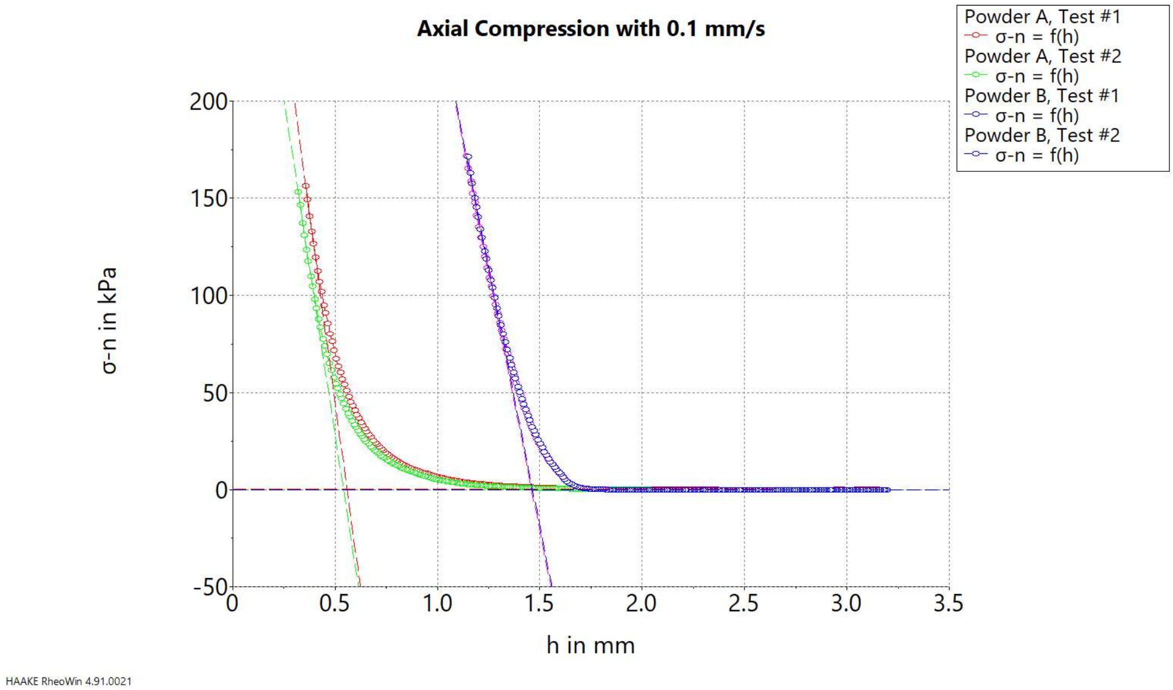

FIGURE 2: Normal stress (σ-n) as a function of the gap height during compression of the powders with 0.1 mm/s at 20 °C. The results of the double measurement of both samples show good reproducibility of the measurement results.

The compression tests results show a clear difference between the two powders (**Fig. 2**). Right from the start, Powder A showed less resistance against compression. At a normal stress of 3.2 kPa (1 N), Powder A was already compressed to 50 % of its original height, whereas Powder B was at 72 % of its original height (**Table 1**). At the end of the test, at 153 kPa (48 N) the sample height of Powder A was only 15 % of its original height compared to 51 % for Powder B, a factor of 3.5 between both samples.

TABLE 1: Evaluation of the compression tests based on sample height (h) and tangent intercept (TI). These results illustrate the better compressibility of Powder A in comparison to Powder B.

	Powder A #1		**Powder A #2**		**Powder B #1**		**Powder B #2**	
	mm	**%**	**mm**	**%**	**mm**	**%**	**mm**	**%**
h(3.2 kPa)	1.19	52	1.11	48	1.65	72	1.65	72
h(153 kPa)	0.36	16	0.32	14	1.17	51	1.18	51
TI	0.56	24	0.54	23	1.46	63	1.46	63

As an alternative evaluation method, tangents have been fitted to the linear parts of the σ-n curves and their intersect (TI) was calculated (dotted lines in Feil! Fant ikke referansekilden.). In contrast to the arbitrary selection of individual data points for comparison, the entire dataset of one measurement is used here. Based on the TI-value, there is a factor of 2.7 between the compressibility of the two powders.

The results of the amplitude sweeps show a plateau of G’ around 2,000,000 Pa (**Figure 3:**) for both powders. Based on these plateaus, the end of the linear viscoelastic range (EoLVR) was calculated at a 10 % deviation from the plateau value.

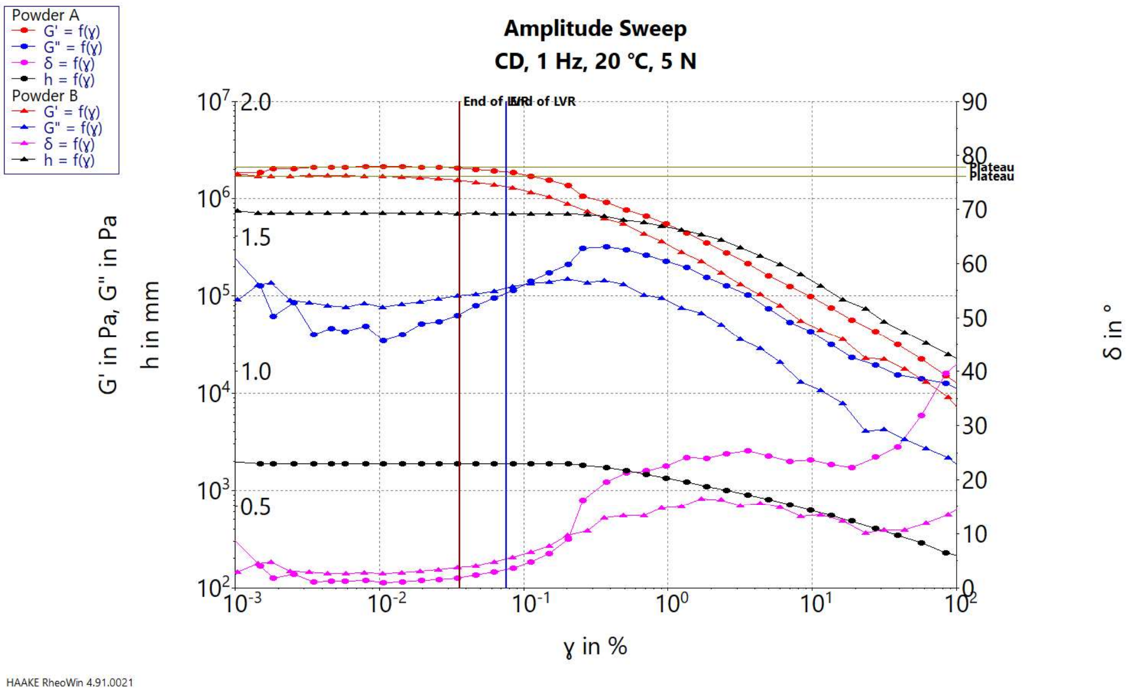

FIGURE 3: Results from the amplitude sweeps at 20 °C plotted over the deformation γ in %. In the non-linear range, the samples started to consolidate further (black curves).

The EoLVR of Powder A was calculated at 0.08 % deformation, whereas the LVR of Powder B already ended at 0.04 % (**0**). This difference became more pronounced when comparing the corresponding shear stresses. The LVR of Powder A ended at 1425 Pa. For Powder B the LVR ended at 549 Pa, a factor of 2.6 lower.

On top of the rheological information, this test also showed that the samples started to compact further after the oscillatory deformation reached certain values in the non-linear range (black curves in **Fig. 3**).

The results of the stress ramp tests were evaluated in the same way as conventional yield point determinations. The resulting deformation was plotted as a function of the applied stress in a double-logarithmic plot and tangents were fitted to both linear parts of the resulting curve. The intersect of these tangents was calculated and used to describe under which conditions the powder started to flow. Although the consolidation of the samples and the axial force applied during the measurements were identical to those used for the amplitude sweep tests, the difference in the results of the two powders was less pronounced for the stress ramp test.

For Powder A, the tangent intersect was at a shear stress of 4242 Pa. For Powder B, the intersect was at 3063 Pa, which is lower only by a factor of 1.4. The corresponding deformation values were almost identical at 0.32 % and 0.33 % (**Figure 4:**).

Here, the samples started to further compact directly after the applied shear stress exceeded the stress value at the tangent intercept (black and grey curves in **Figure 4:4**).

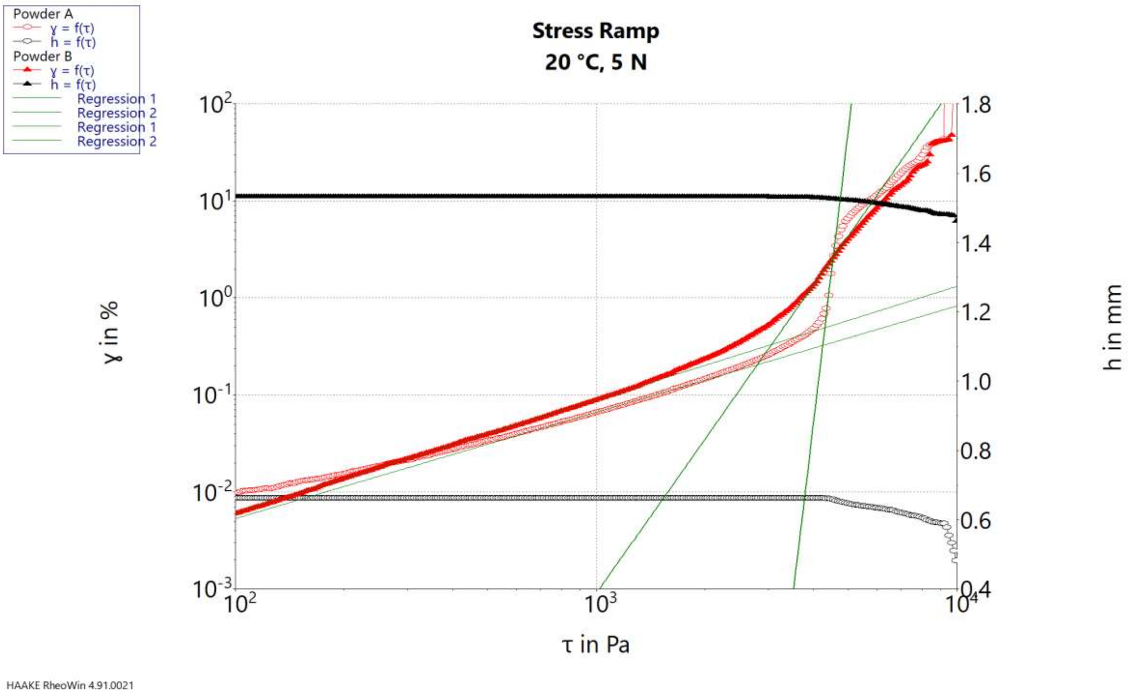

FIGURE 4: Results of the stress ramp tests on both powders at 20 °C and with a normal stress of 15.9 kPa (5 N). The samples started to further consolidate at stresses above the tangent intersect.

TABLE 2: Evaluation of amplitude sweeps and stress ramps. The deformation and the shear stress values at the EoLVR and the tangent intersect (TI) have been calculated.

	Amplitude Sweep			**Stress Ramp**	
	G'(Plateau) / Pa	**γ(EoLVR) / %**	**τ(EoLVR) / Pa**	**γ(TI) / %**	**τ(TI) / Pa**
Powder A	2.08×10^6	0.076	1425	0,322	4242
Powder B	1.69×10^6	0.036	549	0,330	3063

CONCLUSIONS

Two powders have been compared with different testing methods using a HAAKE MARS 60 Rheometer equipped with a parallel plate geometry and a shallow cup ring.
The compression tests showed excellent reproducibility and enabled the comparison of the bulk volume of both powders. The amplitude sweeps and the stress ramps were used to determine the force necessary to make the powders flow. Both measurements were able to distinguish between conditions where the powder reacted like an elastic solid and conditions where the powder started to flow. Between these two tests, the amplitude sweep seemed to be the more sensitive one since it showed a larger difference between the powders.
Except for the shallow cup ring, only standard accessories were used to characterize the powders in a very cost-effective way with regards to their compacting and flow behaviours.

REFERENCES

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