Powder rheology of steel powders for additive manufacturing

O. Lyckfeldt O, Swerea IVF AB, PO Box 104, SE-431 22 Mölndal, Sweden. ola.lyckfeldt@swerea.se

The recent development of advanced rheometers for dry powders characterization has opened opportunities to accurately determine a variety of powder properties to be related to function in specific powder processes. In this study, steel powders aimed for additive manufacturing (AM) were exposed to various types of measurements such as basic flow ability, compressibility, aeration response and shear using a powder rheometer. The results showed that different batches of the same quality of stainless steel (316L) powders, with similar particle size distribution, vary significantly regarding the dry flow properties with critical impact on the function in AM. Differences in specific surface areas indicated variation in particle shape/roughness that could be correlated to rheometer data. Low compressibility, high aeration response and low shear resistance related to low degree of powder cohesivity was identified as favorable characteristics. Hence, powder rheology provides a powerful tool for powder quality control and to confirm processing performance.

Introduction

Powder properties within the powder metallurgy area vary depending on the manufacturing process and type of application. Typically, coarser water atomized metal powders are used for pressing whereas finer gas atomized powders are used for metal injection molding (MIM) [1,2]. For pressing, the flow of the powder in dry state is crucial for the feeding and tool filling operations, whereas in MIM the dry flow ability is limited to the loading of powder into the feedstock preparation step. Conventionally, metal powder properties are expressed in terms of chemical composition and particle size distribution. Hall flowmeter is occasionally used to measure powder flow performance, but provides only limited information about the flow properties and is not suitable for many, especially finer, powder types [3]. Besides particle size distribution, other properties such as particle shape, surface roughness as well as surface chemistry can have dramatic effects on the flow performance of a powder and determine its suitability for a specific application. However, until recently there has been a lack of methods to adequately characterize the flow properties that can display the influence of all these powder aspects and make it possible to predict the processing performance.

Additive Manufacturing (AM) in the powder metallurgy area has increasingly gained interest and the various methods have developed towards better accuracy and production capacity [4,5]. The non-mold flexibility allows shaping of individually designed components with features that are difficult or impossible to realize with conventional powder shaping methods. In shaping of metallic components by 3D ink jet-printing technologies, MIM powders are typically used due to the similarity regarding shaped density and sintering requirement. However, in contrast to MIM, the powder is processed in dry state and, consequently, the dry flow characteristics are of crucial importance. Component built-up is conducted by deposition of thin layers (40-45 µm) of powder combined with an ink-jet printing operation after each layer deposit. This requires an outflow to an even and dense powder layer, not prone to move at the deposit of a subsequent layer. In order to verify the powder properties suitable for this process, more sophisticated characterization methods appear necessary.

In this study a powder rheometer (FT4, Freeman Technology, UK) was used to characterize various flow properties of stainless steel powders. This rheometer enables measurement of axial and rotational forces during axial and rotating movement of a blade (Figure 1) within the powder or by applying a shear head (Figure 2) on top of the powder bed [6]. In the latter case a shear plane is created as powder is locked in between the blades of the cell and yield stresses can be measured. Further, a permeable piston is used to measure the degree of powder compressibility as well as the bed permeability when a controlled air flow is applied from the bottom. The effect on the flow ability of the powder at air application, even to fluidization, can also be characterized. An exact volume of the powder is loaded via a sample dividing tool within the measure cell and the inbuilt balance enabling measurement of the bulk density. In all measurements, an initial conditioning cycle with the blade is conducted to remove stresses and entrapped air to ensure repeatability and equality in sampling. Hence, dry powder properties in terms of packing performance, flow energy in conditioned, consolidated or aerated state as well as shear resistance could be defined.

Gas-atomized 316L stainless-steel powders of MIM quality, aimed for AM by 3D ink-jet printing technique, were exposed to rheometer measurements in this study. The results were related to the particle size distribution, the specific surface area and the reported function in a 3D printing machine.

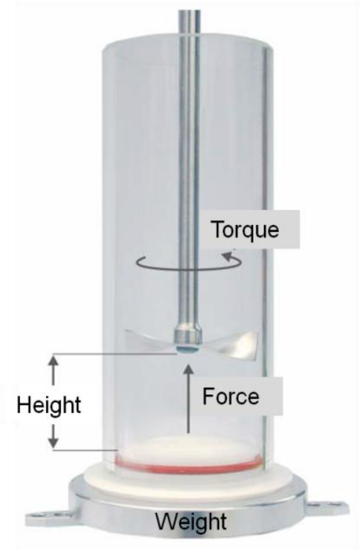

Fig 1: Illustration of blade set-up

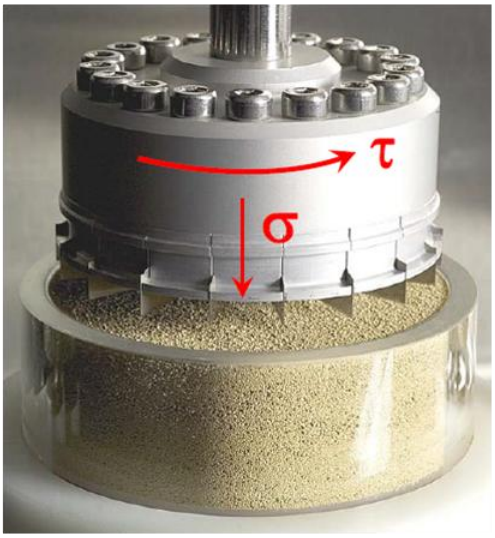

Fig 2: Shear cell configuration

Material and Experimental

Four powder batches of 316 L MIM quality (labeled A, B, C and D) from the same producer were evaluated. Particle size distribution was measured with x-ray sedimentation (Sedigraph, Micromeritics, US) and the specific surface area was measured with the BET method (Gemini 2360, Micromeritics, US). The powder flow and bed properties were characterized in the FT4 powder rheometer. Prior to measurement, the powders were dried at 70°C for at least 12 hours and then kept in a desiccator for cooling to ambient temperature to prevent moisture uptake. Measuring vessels with a diameter of 25 mm with various heights were used for measuring packing performance, flow energy, compression properties, aeration impact, powder bed permeability and yield stresses by shear cell test. For each individual measurement, a new powder sample (10-35 ml) was applied.

The application performance of the powders was tested in a 3D-printing machine (Digital Metal AB, Sweden) in terms of outflow performance, supported by vibration, when a thin (42 micron) layer of the powder was applied via a hopper with 1 mm gap. The powders were simply reported as good, less good or bad based on the out flow ability from the hopper, the evenness in thickness of the applied powder layer and the tendency to move at subsequent layer application.

Results and Discussions

Figure 3 shows the results from the particle-size measurements displaying very similar size distribution for the different powders with only a slight deviation for powder D with less of fines. From this it might be tempting to conclude similar powder flow performance. However, Table 1 shows significant differences in specific surface area (SSA) of the powders as well as the performance in AM.

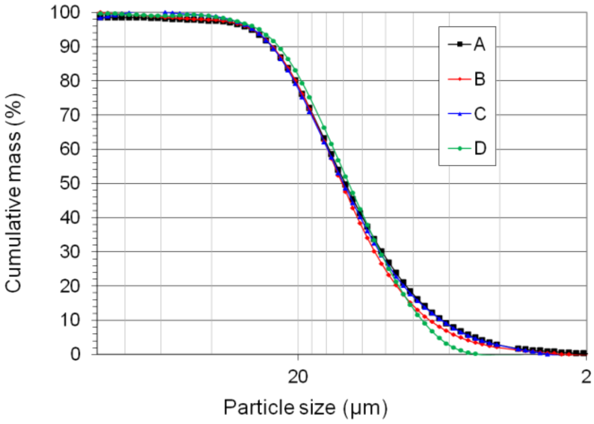

Fig 3: Particle size distributions measured with sedigraph.

Table 1: Specific surface (BET) area and the reported performance in AM of the powders.

Powder	SSA (m^2/g)	Performance in AM
A	0.09	Good
B	0.10	Good
C	0.16	Less good
D	0.22	Bad

The function in this layering process was reported to be good for A and B with the lowest SSA, less good for C with intermediate, and bad for D with the highest SSA. The high surface area of powder D suggests more irregular particle shape, more frequent presence of particle satellites and/or rougher surface texture of the particles. Powder C also showed higher BET-area than A and B indicating higher particle roughness/irregularity than these powders which showed to work-out properly in the process.

Table 2 and Figure 4 show the results from the flow energy measurements conducted with the blade moving downwards (confined powder) and upwards (unconfined) in the powder bed. In between each of the 8 tests, a conditioning cycle was conducted. The level of flow energy expresses the force required to move the powder and the stability index shows how stable the flow properties are. Powder D deviated in both these respects compared to the other powders. However, high flow energy does not necessarily mean that the powder flows badly as a higher bulk density (CBD) requires higher force to move the powder and, therefore, results in higher flow energy. But in this case, powder D displayed a low bulk density as well, strengthening the impression of bad flow performance.

Table 2: Data from flow energy measurements

Powder	SI	SE, mJ/g	CBD, g/ml
A	1,11	2,42	4,63
B	0,98	2,18	4,71
C	1,00	2,66	4,68
D	1,18	4,84	4,42

SI = Stability index
SE = Specific Energy
CBD = Conditioned Bulk Density

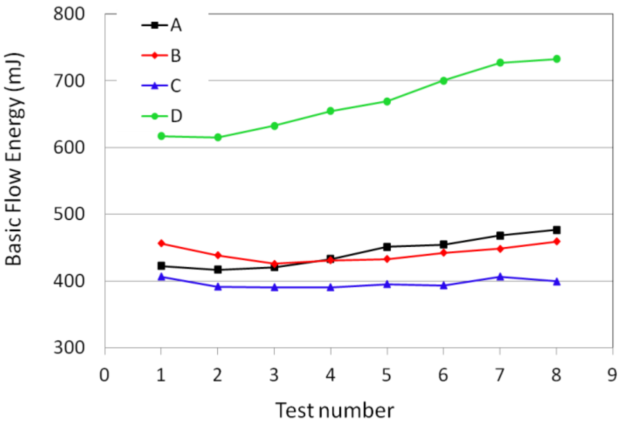

Fig 4: Repeated flow energy measurements with a conditioning cycle in between each measurement.

The specific energy (SE) reflects the flow resistance in the upward movement of the blade and is also related to the CBD. As the powder can be considered unconfined in this operation, a high SE indicates that the particles are strongly attached to each other, e g displays high cohesivity. Here, powder C only showed slightly higher SE than powder A and B whereas powder D displayed a much higher value. Powder D also showed the less stable performance with the largest deviation from 1 of the stability index. This indicated more changes in the powder bed structure along the repeated agitating measurements that can be caused by break down of particle clusters and a resulting bulk density increase. In this respect, powder B and C showed the highest degree of stability with SI values of 1 or near.

Figure 5 shows how the various powders were compressed at increased loading with a clear difference between powder A and B vs powder C and D. High degree of compressibility is an indication of high degree of powder cohesivity as certain stress is required to overcome the adhesion between the particles whereas a non-cohesive powder already is well compacted prior to normal stress application. In practice it means that a more compressible powder is more prone to compact when exposed to compressive forces in a process and can cause problems with disturbed flow performance.

Figure 6 shows the pressure drop over the powder beds at air application. The air permeability depends on the pore size and shape distribution which in turn depends on the particle size distribution and packing as well as compressibility properties of the powder when normal stresses are applied. Powder B and C showed lower degree of permeability at low applied normal stress and less permeability change at increased compressing stress than powder A and B. Although, the latter powders showed high bulk densities the air obviously passing through the beds much easier than in the case of powder B with similar CBD. This indicates differences in particle shape/texture giving rise to variation in the pore structures, affecting the possibility for air to pass. Powder D showed a high pressure drop despite of the low bulk density suggesting even more negative influence of unfavorable particle shape/texture.

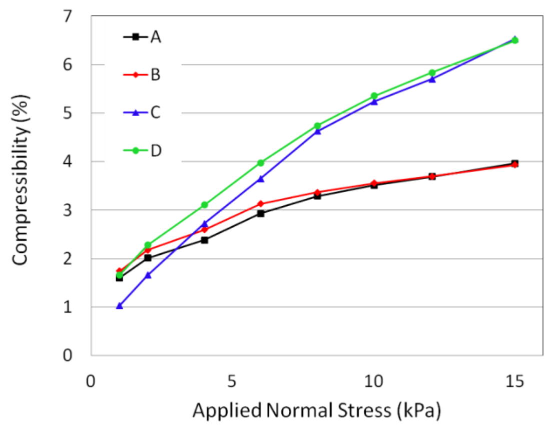

Fig. 5: Degree of compressibility vs applied normal stress.

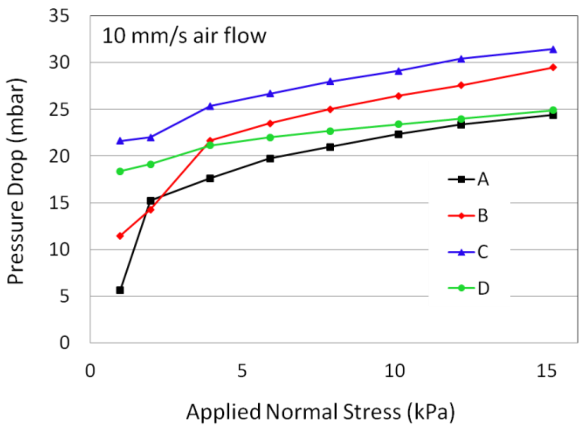

Fig. 6: Pressure drop over the powder bed when applying 10 mm/s air flow at various normal stress.

Figure 7 shows how increased air flow influenced the flow energy of the various powders. It is obvious that powder A and B were more affected by the aeration by stronger decrease of the flow energy with increased air flow. This can be interpreted as the particles in these powders could more easily be separated by the air flow than in the case of powder C and D where stronger cohesivity resulted in less response in flow energy to the air flow.

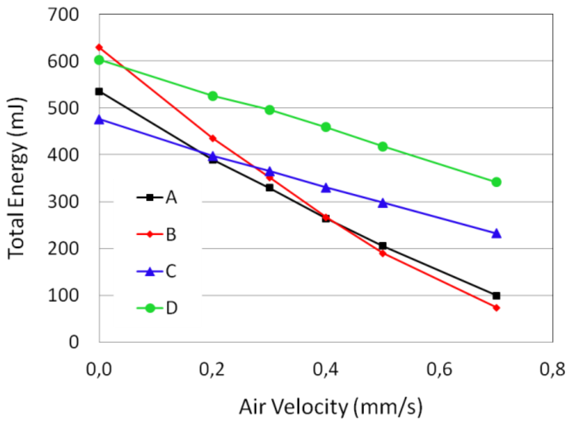

Fig. 7: Flow energy at increased air flow.

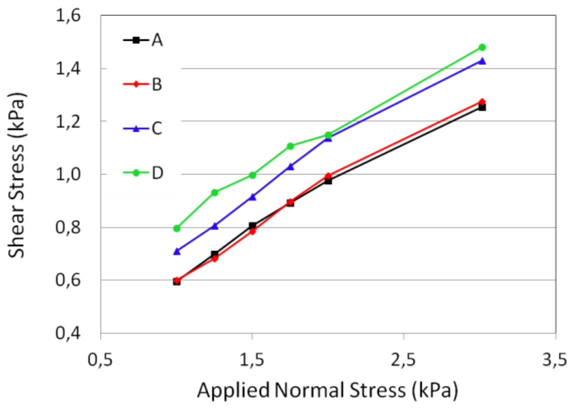

Fig. 8: Shear (yield) stress at various normal forces after a pre-compaction of 3 kPa.

Figure 8 shows the results from the shear cell measurements conducted after a 3 kPa pre-compaction of the powder bed. The level of shear (yield) stress at applied normal force reflects the degree of friction and cohesion between the particles and gives an indication of the difficulty to induce flow. In this case powder A and B clearly displayed lower values than C and D, in consistency with all other presented data. From the yield locus curves in the figure further data can be derived via Mohr circle stress analysis. Among these, table 3 shows the degree of cohesion, the yield stress in unconfined state, as well as a function expressing the flow ability [7]. The cohesivity of C and even more D is confirmed as well as the higher yield stress values required for these two powders.

Table 3: Derived properties from shear cell data based on Mohr circle analysis.

Powder	Cohesion, kPa	UYS, kPa	FF
A	0,22	0,63	6,62
B	0,19	0,56	7,57
C	0,27	0,83	5,29
D	0,46	1,31	3,53

UYS = Unconfined Yield Stress
FF = Flow function

The flow function expresses the fluidity of the powders with a higher value for better flow [8]. Together with this and all other results, powder B showed the overall best flow performance.

Conclusions

This study has demonstrated how powder rheology can be used to define flow properties of metal powders, specifically for use in additive manufacturing but could be applicable as a tool for powder characterization versus function in most powder metallurgy processes. Although, the particle size distribution of the evaluated powders was similar, the flow properties were shown to vary significantly. Due to higher degree of powder cohesivity, the compressibility becomes higher, the aeration effect becomes less pronounced and the yield stress at shear becomes higher. Two of the powders (A and B) showed superior flow performance compared to the others, most probably due to less particle irregularities and/or surface texture indicated by the lower specific surface area. Regarding the AM application, it can be concluded that the degree of cohesion has to be sufficiently low, allowing the powder to easily reach a high packing density, can flow properly on demand but not at the shear forces induced when applying a subsequent powder layer. Consequently, the flow energy has to be high enough but driven by a high tap density and not by cohesive forces.

At validation of rheological results, it is important to have knowledge about the impact of various powder properties and, not at least, relate the results to the function in the specific process. Then powder rheology can be a powerful tool for quality verification for the powder manufacturer as well as functionality control of new batches of powder for the user.

Acknowledgements

This work was part of the EU-project Hyproline (http://www.hyproline.eu), “High performance Production line for Small Series Metal Parts”, with the aim to develop rational production of customized and innovative metal products with high accuracy.

References

[1] Handbook of metal injection molding, Woodhead Publishing Series in Metals and Surface Engineering No. 54, Ed. D.Heaney, Penn State Unviv., USA, 2012.
[2] R.M German, Powder Injection Molding, Metal Powder Industries Federation (MPIF), 1990.
[3] Hall flowmeter, standard ISO 13320:2009(E).
[4] K.V. Wong and A. Hernandez, “A Review of Additive Manufacturing”, IRSN Mechanical Engineering, Vol 2012, pp , 2012.
[5] T.J. Horn and O.L. Harrysson, “Overview of current additive manufacturing and selected applications”, Sci Prog. 2012;95 (Pt 3), pp 255-82, 2012.
[6] R. Freeman, “Measuring the flow properties of consolidated, conditioned and aerated powders — A comparative study using a powder rheometer and a rotational shear cell”, Powder Technology, Volume 174, Issues 1–2, pp 25-33., 2007.
[7] A. W Jenike, *Storage and Flow of Solids*, University of Utah, Bulletin 123, 1964