Introduction to a new standardized test method for powder mixes, the Gustavsson flowmeter funnel

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Abstract
The Hall flowmeter funnel according to ISO 4490 is a well-established test method used in the PM industry for decades. The method is recommended and works well for metallic powders without organic additives such as lubricants and graphite. The Hall funnel is also widely used for determination of apparent density and flow of metallic powder mixes including organic additives. However, a problem is that powder mixes, especially mixes with high content of lubricant, graphite and/or other fine particulate additives, will not always flow freely through the funnel. To overcome this, a new funnel with a 30° cone angle has been developed. The new funnel enables mass flow so "rat holes" leading to flow stops can be eliminated. The new flowmeter funnel, called Gustavsson, is now standardized according to ISO 13517. In this paper a comparison between the Hall and Gustavsson funnels is presented. Different types of metal powders and metal powder mixes have been evaluated and will be reported.

Introduction
A pre-requisite for production of sintered parts is free flowing metal powder mixes. Good flow behaviour is required in order to have high productivity and consistency in the compaction of parts. The Hall flow method is widely used in the PM industry for characterization of powders and was established as a standard by MPIF in 1945. It is also an international standard, namely ISO 4490:2008.

The principle of the Hall flow method is very simple: 50 g of powder flows through a funnel and the time it takes is registered as the flow rate. The funnel has a standardised geometry with 60° cone angle and ø2.5 mm outlet hole and is calibrated by a reference powder "Chinese Emery". A great advantage of the method is its simplicity and the low cost of the required test equipment.

A funnel with 5 mm hole diameter, the Carney funnel, also exists in MPIF standard 28 for measuring apparent density. This funnel is sometimes used to measure flowability of powders which do not flow freely through the Hall funnel. With the large outlet hole the time for 50 g to flow out of the funnel is very short, which results in very little discrimination between powders.

There are arguments held against the Hall flow method. One is that the method is not relevant for charactering the filling of a tool die. For this reason attempts have previously been made within ISO TC119/SC2 to develop and standardize a method for simulating filling of press tools [1]. That work resulted in the conclusion was that it was not possible to develop a method to characterize die filling behaviour which would be generally suitable for the various metal powder mixes in common use.

Another problem that has been identified with the Hall flow method is that PM powder mixes, especially mixes with high content of lubricant, graphite and/or other fine particulate additives, will not always freely flow through the funnel. There also appears to be a random element in the occurrence of "no-flow" mixes. With this background the idea of a modified funnel for flow measurement of PM mixes arose. The modified funnel has a steeper cone angle of 30° instead of 60°, to ensure that the powder flows by mass flow.

The 30° cone angle funnel has now been established as ISO standard ISO 13517 "Metallic powders – Determination of flowrate by means of a calibrated funnel (Gustavsson flowmeter)"

In this paper a comparison between the Hall and Gustavsson funnels is presented. Three aspects of measuring the flow with the two funnels are covered: influence of the powder mix composition on the measured flow rate, occurrence of flow stops in production control and results of gauge R&R studies.

Apparatus

In Figure 1 the design of the Gustavsson flowmeter is presented. The design is very similar to the Hall flowmeter, with the main difference that the cone angle of the funnel is 30° instead of 60° as in the case of the Hall funnel. An important aspect of flow measurement is the means for calibration of the funnels. The Gustavsson funnel uses the same calibration powder, Chinese emery, as is used for the Hall funnel. The nominal flow time with the calibration powder is also the same for the two flow meters: 40 s/50 g.

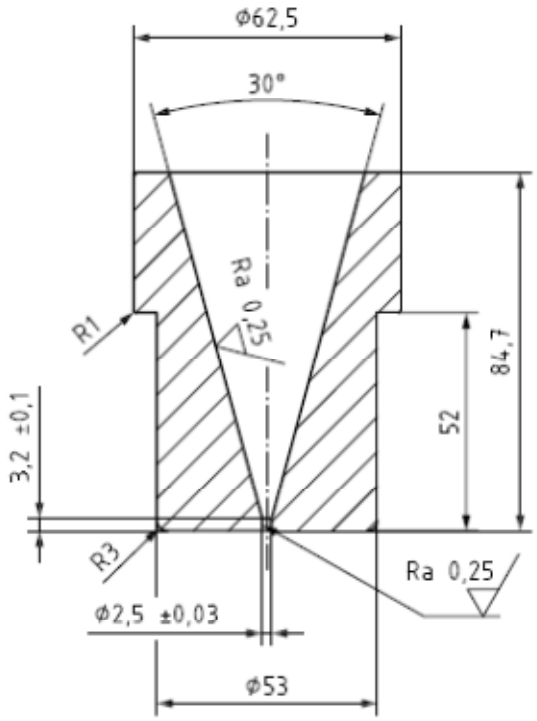


Figure 1: Design of Calibrated funnel (Gustavsson flowmeter), dimensions in millimetres

By having a steeper cone angle the Gustavsson flowmeter works as a mass flow hopper which is often not the case for the Hall funnel when testing press ready mixes. This is illustrated in Figure 2 where a mix with composition according to Mix A, see Table 1, was tested in the two types of funnels.

In the Hall funnel the powder is flowing by funnel flow and the powder only flows in the centre of the funnel and on the upper powder surface. The rest of the powder is stagnant which increases the risk of bridge formation. Flow stop may occur if a fully developed “rat hole” or a bridge is formed in the powder.

In the Gustavsson funnel the powder is flowing by mass flow and the complete powder volume is under continuous shearing and bridges will not form. As the powder is flowing by mass flow no “rat holes” will be formed.

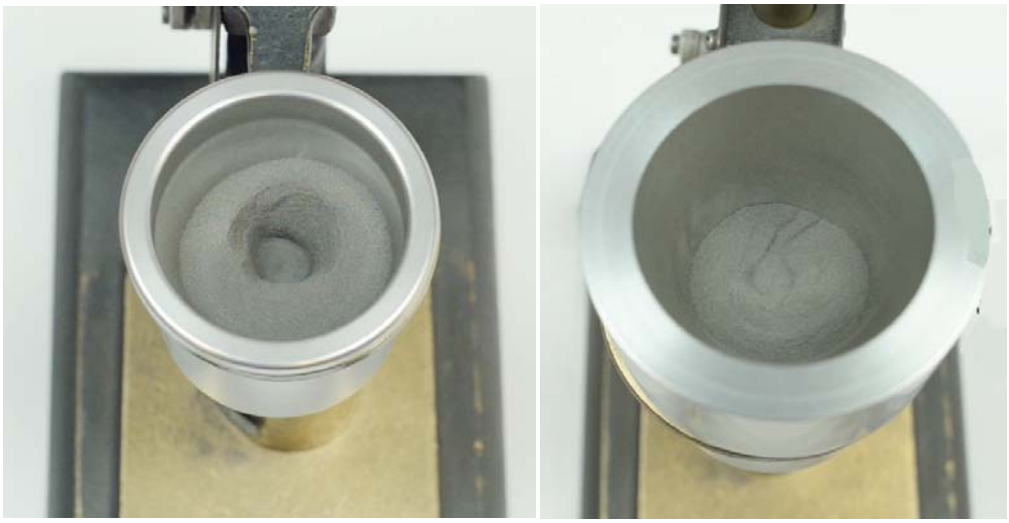


Figure 2: Flow patterns in Hall Flowmeter (Left) and the Gustavsson flowmeter (Right)

Experimental

Evaluation of different powders

To assess performance of the two funnels a number of mixes have been tested. These mixes had the following compositions:

- ASC100.29 + X% C-UF4 + X% Amidewax (designated A0.5 – A1.0)
- ASC100.29 (varying fines content) + 0.8% C-UF4 + 0.8% Amidewax (designated A0 – A40)
- ASC100.29 + X% C-UF4 + X% Zn-st (designated Z0.5 – Z1.2)

The intention of these mixes with varying fines content and varying graphite and lubricant contents was to create series of mixes with incrementally decreasing flowability.

Six mixes were made with graphite and Amidewax contents ranging from 0.5% up to 1.0%. In each mix the amounts of graphite and Amidewax were the same. For example mix A0.5 contained ASC100.29 + 0.5% C-UF4 + 0.5% Amidewax. Eight mixes were made with graphite and Zn-st content ranging from 0.5% up to 1.2%. The amount of graphite and Zn-st in each of these mixes were the same.

Five mixes were made with varying amount of fine particles (<45 μm), ranging from 0% to 40%, with increments of 10%. These mixes are designated A0 to A40 where the number is the fines content in per cent.

ASC100.29 is water atomized pure iron powder, C-UF4 is a natural graphite powder and Amidewax and Zn-st are two of the most commonly used lubricants in press ready mixes.

Occurrence of flow stop of difficult production mixes

The occurrence of flow stops with the two types of funnels will be exemplified with QC data from production of press ready mixes at Höganäs AB. Statistical data have been collected from the quality control of produced lots over a time period of about six months where each lot has been tested with both funnels. Flow measurement with the two funnels was carried out by an automated test station (see Figure 3 for picture of the equipment). By fully automatic measurement there was no operator influence on the measured results.

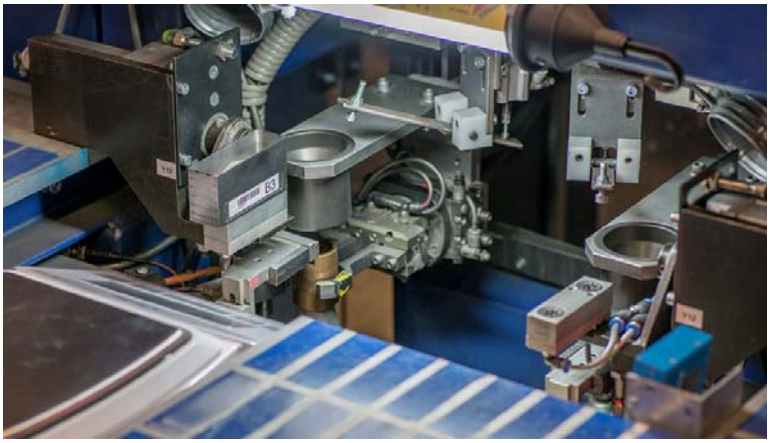


Figure 3: Equipment used for automatic measurement of flow

Flow stop statistics are presented for the mixes in Table 1. These three mixes have been selected based on history of being "difficult" mixes with recurring flow stops. Besides statistic on flow stops the average flow time and standard deviation between lots will be presented.

Table 1: Mix compositions

Mix	Composition
A	Distaloy AB + Graphite + MnX + Lube M + Binder
B	AHC100.29 + Cu-100 + Graphite + Zn-st
C	Distaloy AB + MnS + Graphite + Amidewax

MSA – Gauge R&R study

Investigations of Repeatability and Reproducibility have been conducted for both funnels on press ready mixes in accordance with guidelines in [2]. The Range method was used for the Hall funnel and the Average and Range method was used for the Gustavsson funnel.

Results and discussion

Evaluation of different powders

Flow rates for mixes with increasing contents of graphite and Amidewax are presented in Figure 4, and in Figure 5 flow rates for mixes with increasing contents of graphite and zinc stearate are presented. There are a number of observations to be made from the results presented in these figures. First it is obvious that flow times are much longer with the Gustavsson funnel compared to the Hall funnel. Mass flow is the preferred flow pattern in powder handling but it does not mean that the flow rate is faster for mass compared to funnel flow. The calibration powder for the funnels "Chinese emery" will flow by mass flow in both funnels and also with the same flow time in both funnels. Thus the difference in flow time will arise with deteriorating flowability of press ready mixes where the flow pattern in the Hall funnel will change to funnel flow while the flow pattern of the Gustavsson funnel will still be mass flow.

It can also be noted that the influence on measured flow rate from additive content is much stronger with the Gustavsson funnel. Finally, with the Hall funnel mixes A1.0, Z1.1 and Z1.2 did not flow through the Hall funnel but did flow through the Gustavsson funnel.

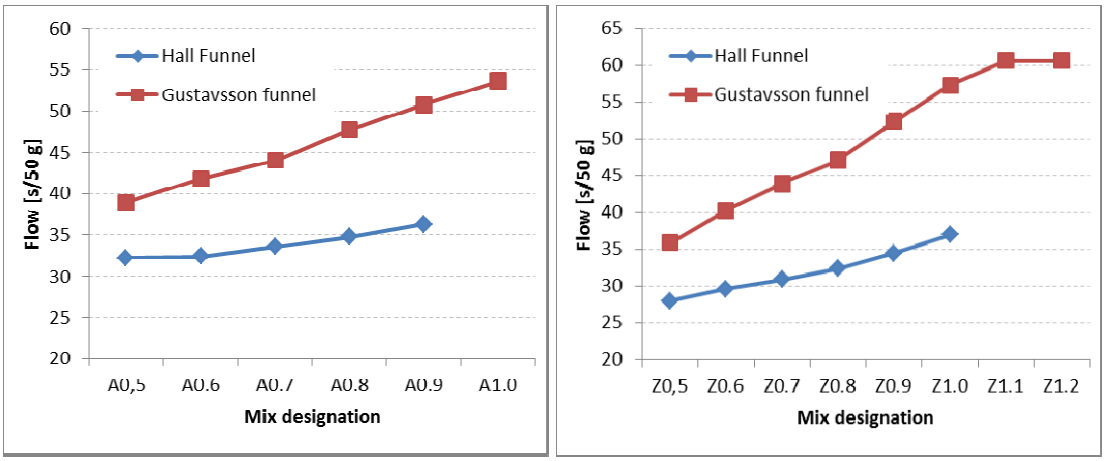


Figure 4 and 5: Flow rate of mixes with increasing contents of graphite and Amidewax (left) and graphite and zinc stearate (right)

In Figure 6 flow rates for mixes with increasing contents of fines fraction in the base powder are presented. The results in this experiment show exactly the same patterns as for the mixes with increasing additive contents. The influence of the fines content is much stronger with the Gustavsson funnel and the mix with the highest fines content only flowed when tested in the Gustavsson funnel. From these results it can be concluded that the Gustavsson funnel is more sensitive to variations in the mix composition and that it can be used for testing, from a flow perspective, more difficult mixes, here exemplified by high additive and fines contents.

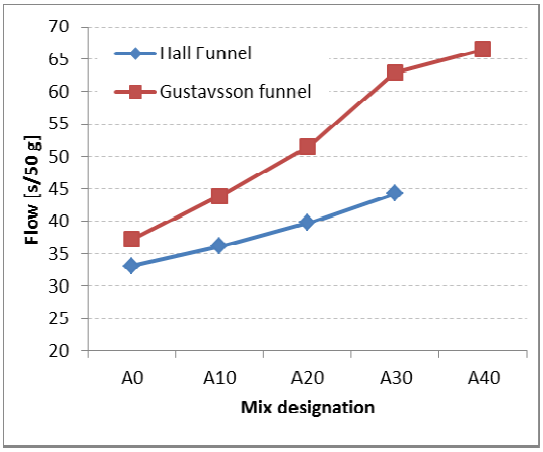


Figure 6: Flow rate of mixes with increasing fines (-45 μm) contents of the base powder

Occurrence of flow stop of difficult production mixes

Table 2 presents results from QC on flow measured with the two types of funnels. From the data on the percentage of lots with flow stops in the test 24 hours after the mix was blended, there was a major improvement from when using the Gustavsson funnel. If flow stops occur in the QC, a new sample of the lot will be taken and a new analysis has to be carried out.

Flowability of press ready mixes generally improves by time. Due to this time factor as well as the random element in the occurrence of flow stops with the Hall funnel, most lots will eventually flow after one or two re-samplings.

Table 2: Mix compositions

		Hall funnel			Gustavsson funnel		
			Flow (s/50 g)			Flow (s/50 g)	
Mix	# lots tested	% lots with flow stop	Mean	Std. dev.	% lots with flow stop	Mean	Std. dev.
A	97	12%	30.4	1.1 (3.5%)	6%	34.9	2.0 (5.6%)
B	55	55%	37.4	1.2 (3.2%)	9%	55.8	2.9 (5.1%)
C	37	54%	37.1	0.9 (2.3%)	3%	49.3	2.1 (4.3%)

In Table 2 it can also be seen that flow time is significantly longer with the Gustavsson funnel and between lot variations, expressed as one standard deviation, is larger both in seconds and as a percentage of the average flow time.

MSA – Gauge R&R study

In Table 3 results from gauge R&R studies for the flow measured by the two funnels are presented. The numbers for the GR&R represent 5.15 standard deviations. Assuming that the variation is normally distributed 99% of all measured results should fall within the range of the GR&R. Although slightly different methods were used to assess the two funnels the results should be comparable. The results show that there is no significant difference in the scatter of the test method when measuring flow with the two funnels.

Table 3: Result of Gauge R&R studies

	MSA Method	GR&R [s/50g]
Hall Flow	Range method	1.5
Gustavsson flow	Average and Range method	1.4

From this it can be concluded that the increased scatter between lots when using the Gustavsson funnel, presented in Table 2, is not due to larger scatter of the test method. The explanation can be that the Gustavsson funnel is more sensitive to between lot variations of the powder mixes than the Hall funnel.

Conclusions

- Press ready mixes will generally flow by mass flow in the Gustavsson funnel whereas the flow pattern in the Hall funnel is funnel flow
- Flow times are longer with the Gustavsson funnel compared to the Hall funnel
- Scatter of the test method (GR&R) is comparable between the two types of funnels
- With the Gustavsson funnel the response to variations of the mixes composition, such as contents of additives of fines, is much stronger
- As a consequence, measured variation between production lots is greater with the Gustavsson funnel
- The inclination for flow stop is much smaller with the Gustavsson funnel and the method is applicable for a wider range of compositions. Therefore the method is better suited for characterization of press ready mixes

References

1. E.R. Rice and J. Tengzelius in Powder Metallurgy 1986 Vo.29 No. 3, 183 – 194
2. MSA Fourth Edition, Chrysler group LLC, Ford Motor Company and General Motors Corporation