CHEMICAL ENGINEERING TRANSACTIONS

VOL. 74, 2019

A publication of AIDIC
The Italian Association of Chemical Engineering
Online at www.cetjournal.it

Guest Editors: Sauro Pierucci, Jiří Jaromír Klemeš, Laura Piazza

ISBN 978-88-95608-71-6; **ISSN** 2283-9216

DOI: 10.3303/CET1974066

Characterising Powder Flowability at High Shear Rates by the Ball Indentation Method

Monica Tirapelle[a], Andrea C. Santomaso[a], Colin Hare[b,*]

[a] APTLab - Advanced Particle Technology Laboratory, Department of Industrial Engineering, University of Padova, IT
[b] Department of Chemical and Process Engineering, University of Surrey, Guildford, GU2 7XH, UK
c.hare@surrey.ac.uk

Unreliable powder flow is a major problem during processing of powders. The shear cell is the most widely used method for powders subjected to moderate or high stresses, and under quasi-static conditions, with established methods for designing large bins and hoppers based on the measurement. However, this method is not suitable for measuring the flowability of dynamic systems, such as powder mixing.
Here, the ball indentation method is investigated as a technique for evaluating powders in the intermediate and dynamic regime of flow. The method, which simply consists of dropping a ball onto a cylindrical bed of powder previously consolidated, directly measures hardness, which is related to the unconfined yield stress of the powder by the constrain factor (Hassanpour and Ghadiri, 2007). The impact of the ball on the bed is recorded with a high-speed camera to determine velocity and penetration depth. The shear rate is varied by using a range of indenter materials and sizes, and a range of drop heights. The hardness against the strain rate is considered for several materials.
It was found that the indenter size does not influence the hardness results, which are consistent with the flowability evaluation achieved with the rheometer. Furthermore the hardness, which is independent of the strain rate in quasi-static conditions, becomes shear rate dependent in intermediate regime of flow. Further work is needed to evaluate hardness in the rapid granular flow regime.

1. Introduction

In industrial equipment, such as screw conveyors and shear mixers, reliable powder flow is important for final product quality and for a consistent production rate. Since shear stress is dependent on the strain rate, understanding the flow behaviour of powder beds as a function of the shear rate is particularly important (Pasha et al., 2015). Furthermore, despite the large number of existing methods for measuring the flow properties of bulk powders (Santomaso et al., 2013; Shulze, 2008; Salehi et al., 2017), only a few techniques allow investigation of the flow behaviour of powders in intermediate and dynamic regimes of flow.
The classification of the flow regimes is based on the dimensionless shear rate, $\dot{\gamma}^*$, as proposed by Tardos et al. (2003):

$$\dot{\gamma}^* = \dot{\gamma}\sqrt{\frac{d_p}{g}} \tag{1}$$

where d_p is the mean particle diameter, g is the gravitational acceleration and $\dot{\gamma}$ is the shear rate. According to this classification, the powder is considered to be in quasi-static or "slow-frictional regime" for $\dot{\gamma}^*$ < 0.15; in intermediate regime for 0.25 < $\dot{\gamma}^*$< 3; and in dynamic regime for dimensionless shear rates grater than 3. The boundaries between regimes are still uncertain and not clearly defined (Tardos et al., 2003).
Hassanpour and Ghadiri (2007) proposed a test method for evaluating powder flowability at low compaction level and small scale. The method consists of applying ball indentation on a bed of cohesive powder; therefore the material hardness, H, which represents the resistance of the material to plastically deform, can be measured. They showed that the hardness measured by ball indentation correlates well with the unconfined yield stress through the constraint factor, C, that depends on the mechanical properties of the material (Pasha

Paper Received: 29 June 2018; Revised: 29 November 2018; Accepted: 10 April 2019

Please cite this article as: Tirapelle M., Santomaso A., Hare C., 2019, Characterising Powder Flowability at High Shear Rates by the Ball Indentation Method, Chemical Engineering Transactions, 74, 391-396 DOI:10.3303/CET1974066

et al., 2013). Once the constraint factor of the material is characterised, the unconfined yield stress can be estimated from hardness measurements.

The present paper is concerned with the study of the rheological behaviour of bulk cohesive powders at higher shear rates. The intermediate regime has been fully addressed by applying the dynamic ball indentation method on three different materials, whose characteristics do not allow reaching shear rates beyond the intermediate regime.

It was found that hardness remains constant for shear rates characteristic of the quasi-static regime, whilst it becomes shear rate dependent in the intermediate regime of flow where not only friction but also collisions between particles becomes significant (Tardos et al., 2003). Anyhow, further works are needed for directly addressing the shear rate sensitivity in the dynamic regime.

2. Materials and methods

The dynamic ball indentation method is carried out on samples of Titanium Dioxide R104, Titanium Dioxide DT51 and Waxy Corn Starch.

The samples are firstly poured into a 50 mL die with 50 mm diameter and then subjected to the conditioning process of the FT4 powder rheometer (Freeman Technology) for achieving a reproducible and homogeneous state. The material is successively pre-consolidated to different pressure levels by the vented piston of the FT4, which moves downward with a velocity of 0.05 mm/s. Once the desired normal stress is reached, it is maintained for 1 minute to avoid any elastic recovery. Finally, the bed of powder such consolidated is subjected to dynamic indentation: a spherical indenter, which is initially held by a vacuum pump, is dropped by switching off the pump and the impact of the ball on the bed is recorded with a high-speed camera.

To vary the shear rate, a range of indenter materials and sizes, and a range of drop heights are needed. Here, sixteen different indenters made of Nylon, Glass, Ceramic and Stainless Steel, and with diameters equal to 3.17, 4.77, 9.52 or 15.87 mm have been used. It is noteworthy that the size of samples and indenters, as well as the distance between the indentation load and the surrounding walls, has been chosen in accordance with the specifications of the operation window analysed by Pasha et al. (2013).

The experimental procedure does not allow the loading/unloading curve nor the maximum indentation load, F_{max}, to be recorded, hence the hardness of the consolidated bulk cannot be calculated as proposed by Hassanpour and Ghadiri (2007). Here, the expression of dynamic hardness, H_d, suggested by Tirupataiah and Sundararajan (1990) is used:

$$H_d = \frac{M\, v_i^2}{2\, U} \tag{2}$$

where M is the mass of the indenter and v_i is the impact velocity. The crater volume, or unrelaxed volume, U, is calculated using the standard geometrical relation for estimating the volume of a spherical cap. To determine the indenter impact velocity and the depth of the penetration, post-process image analysis is performed.

All experiments reported in this work are carried out under ambient conditions of 20–25 °C and relative humidities of 45– 60 %.

3. Results and discussion

In this section the quantitative and qualitative characterization of the investigated powders is discussed before reporting the hardness results obtained with the ball indentation technique.

3.1 Flow properties of the powders

Three materials are tested: Titanium Dioxide (TiO_2) R104, Titanium Dioxide (TiO_2) DT51 and Waxy Corn Starch. The grades of Titanium Dioxide are chosen because of their cohesiveness, whilst the starch because of the bigger particle dimension and hence the possibility to achieve higher shear rates.

All tested materials are characterised by poly-dispersed size distributions. However they differ for size and flow behaviour. The volumetric median diameters, $d_{50,3}$, of TiO_2 R104 and TiO_2 DT51 reported in Table 1 are measured using the Zetasizer (Malvern Instruments); whilst the dynamic image analyser QicPic is used for Waxy Corn Starch. The difference in particle size is needed for investigating a wider range of dimensionless shear rates since $\dot{\gamma}^*$ is dependent on particle diameter (see Eq. 1). However, with increasing size, the powder cohesiveness diminishes. In Table 1 the flow behaviour of the powders based on the flowability index classification proposed by Jenike (1964) and determined with the rotational shear tester of the FT4 powder rheometer for two different levels of pre-shear normal stress (3 and 9 kPa) is reported.

Table 1: powder median diameter, flow factor (ff) at 3 and 9 kPa pre-shear and flow behaviour according to the flowability index classification proposed by Jenike (1964).

Powder	$d_{50,3}$(μm)	*ff* at 3 kPa	*ff* at 9 kPa	Flow behaviour
TiO_2 R104	0.38	0.95	1.01	Not flowing-very cohesive
TiO_2 DT51	1.08	1.64	2.27	Very cohesive-cohesive
W. Corn Starch	30.60	4.64	7.31	Easy Flowing

Because hardness measures the resistance of a material to plastic deformation, the range for indentation load within which a relatively constant value of hardness is obtained has to be identified. Its lower limit is where plastic flow in the bed is initialized; the upper limit may be caused by consolidation during testing, or due to further constraint supplied by the die if the combination of die diameter and indenter size are not appropriate (Pasha et al., 2013). Hence, in order to investigate the range of dimensionless penetration depths (penetration depth divided by indenter radius) where hardness can be considered constant, the FT4 indentation process was performed on previously compacted samples of each material, considering dimensionless penetration depths between 0.1 and 0.9 (Fig. 1). In quasi-static indentation (using the rheometer) the hardness, *H*, is given by the ratio of the maximum indentation load, F_{max}, to the projected area of the penetration, *A*.

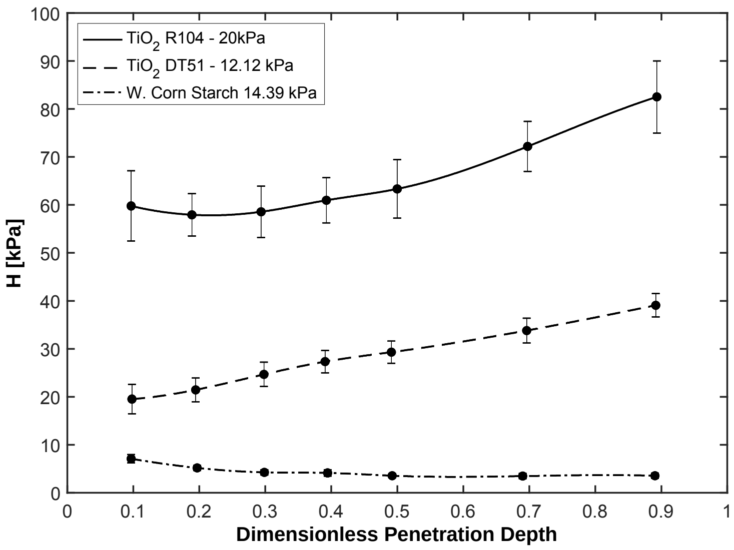

Figure 1: Quasi-static indentation hardness as a function of dimensionless penetration depth.

The experimental results in Figure 1 show that the hardness of TiO_2 R-104 is stable for dimensionless penetration depths ranging from 0.1 to 0.5. For higher values of dimensionless penetration depth, hardness is overestimated. In the case of TiO_2 DT-51, a range of dimensionless penetration depths with relative constant hardness cannot be identified. As it can be seen, the measured hardness rises as the penetration depth increases. For Waxy Corn Starch, hardness is stable in the whole range investigated, with slightly higher values for dimensionless penetration depth smaller than 0.3. From Figure 1, it can also be said that more cohesive powders exhibit higher hardness, meaning that hardness and flowability are correlated.

3.2 Indentation results

The purpose of this study was to investigate the hardness dependency on the dimensionless shear rate.

Figure 2 shows some hardness results obtained with the dynamic ball indentation method by dropping all the sixteen available indenters onto samples of TiO_2 R104 previously compacted to 20 kPa, when the impact velocity of the spheres was 1.99 ± 0.04 m/s. Here, hardness is independent of the shear rate in quasi-static condition and in the uncertain boundary between quasi-static and intermediate regime of flow when using Nylon, Glass, Ceramic and Stainless Steel indenters. Hardness does not increase when $\dot{\gamma}^*$ is increased to approximately 0.25, as predicted by Tardos et al. (2003). In any case it should be noted that the particles tested here cannot be assumed to be perfect spheres with mono-dispersed size distribution, therefore there is uncertainty in the determination of $\dot{\gamma}^*$.

Figure 2 shows that the hardness measured is greater when using the Stainless Steel indenters than other indenter materials. This over estimation of hardness with Stainless Steel indenters is explained in Figure 3 where the same results are shown as a function of dimensionless penetration depth. When drop height and indenter diameter are fixed, Stainless Steel, which is the heaviest material, penetrates deeper in the powder bed, reaching the range of penetration depths where hardness cannot be considered constant anymore (see Figure 1). Figure 3 shows also the quasi-static hardness measurements from which it appears that the two indentation techniques correlate well with each other.

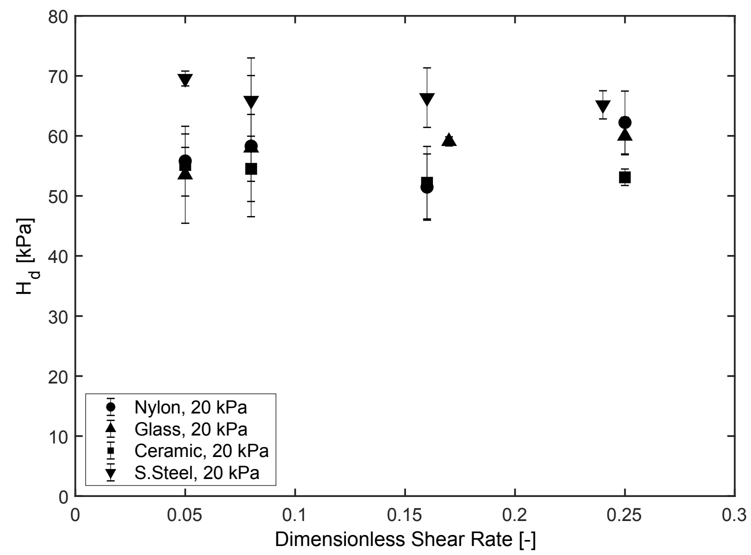

Figure 2: Hardness as a function of dimensionless shear rate for TiO_2 R104 pre-consolidated at 20 kPa. The indenter impact velocity v_i is 1.99 m/s.

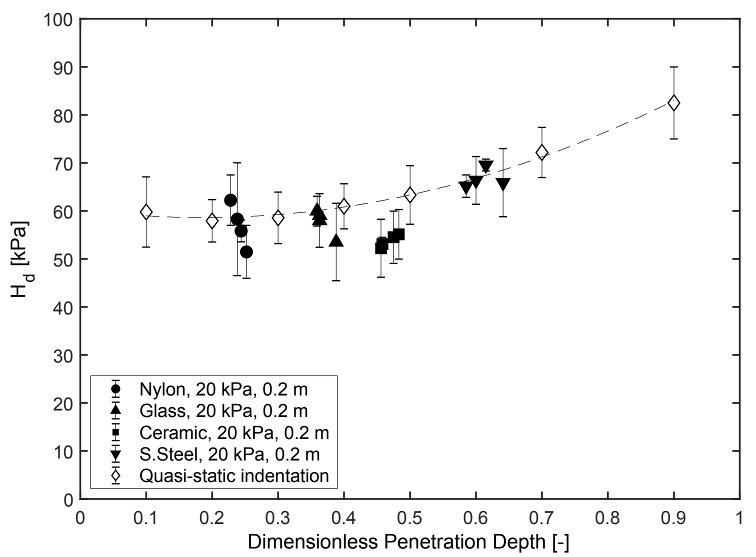

Figure 3: Hardness versus dimensionless penetration depth for TiO_2 R104 pre-consolidated at 20 kPa obtained with the dynamic ball indentation method and the FT4 quasi-static indentation process.

All the sixteen indenters are also used to evaluate the flowability of TiO_2 DT51 pre-consolidated at 12.12 kPa. The impact velocity of the indenters in this case was lowered to 1.40 ± 0.02 m/s in order to achieve an acceptable penetration depth. As Figure 4 shows, hardness markedly increases for $\dot{\gamma}^*$ beyond 0.2. Therefore, hardness is relatively independent of shear rate in the slow frictional flow regime; whilst it becomes shear rate dependent at higher values of $\dot{\gamma}^*$, namely in the transition between the quasi-static and the intermediate, as well as in the lower range of the intermediate regime.

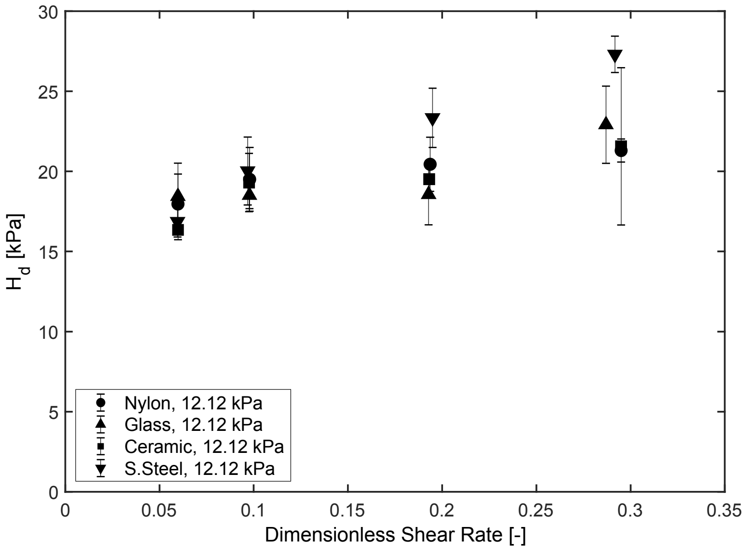

Figure 4: Hardness as a function of dimensionless shear rate for TiO_2 DT51 pre-consolidated at 12.12 kPa. The indenter impact velocity, v_i, is 1.40 m/s.

Further investigations in the intermediate regime have been possible with Waxy Corn Starch because of its larger particle size. However, the operating conditions that could be applied were limited due to the powder flow behavior. In Figure 5 the hardness results achieved with Glass and Ceramic indenters dropped from 0.05 m height (v_i = 1.06 ± 0.04 m/s) and Nylon indenters released from 0.10 m height (v_i = 1.35 ± 0.03 m/s) are reported. In all cases the samples have been previously compacted at 14.89 kPa. In this case, hardness increases rather linearly with the shear rate, meaning that hardness is shear rate dependent in the intermediate regime of flow, where not only friction, but also interactions between particles must be considered (Tardos et al., 2003). The results prove that the ball indentation method can well capture the dynamics of powder flow.

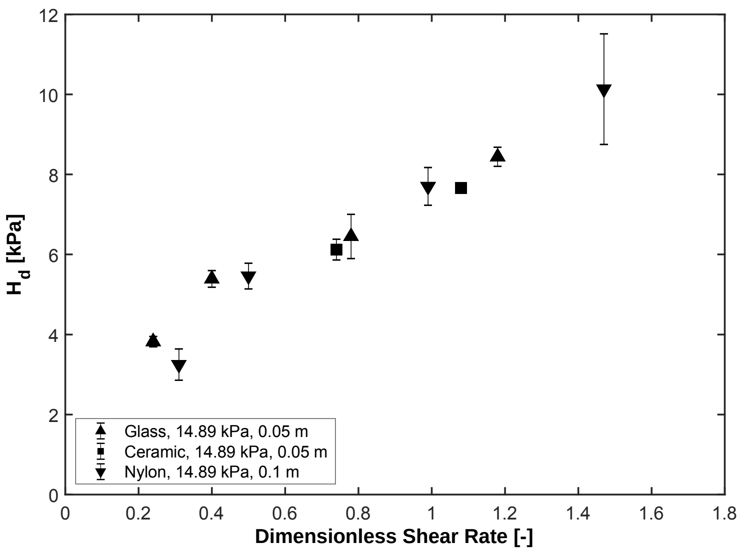

Figure 5: Hardness as a function of dimensionless shear rate for Waxy Corn Starch pre-consolidated at 14.89 kPa. The indenter impact velocities, v_i, are 1.06 m/s and 1.35 m/s respectively for 0.05 and 0.10 m drop height.

To directly address the shear rate dependency of hardness in the dynamic regime of flow, further works should be done. In order to be able to perform indentation at greater values of shear rate, it is recommended to use cohesive powders characterized by larger particles, for instance glass beads made cohesive by silanisation, and smaller indenters.

4. Conclusions

The Ball Indentation Method is experimentally applied on compacted assemblies of TiO_2 R104, TiO_2 DT51 and Waxy Corn Starch. The hardness is evaluated in a range of dimensionless shear rates.

It was found that hardness does not vary with the shear rate in the quasi-static regime of flow since friction between particles is predominant and inter-particle interactions are negligible, as suggested by Tardos et al. (2003).

In the intermediate regime of flow, where inter-particle interactions become more energetic, an increase in hardness with the shear rate was observed. However, the threshold dimensionless shear rate from which hardness starts becoming shear rate dependent cannot be universally defined so, for instance, if this limit is 0.20 for TiO_2 DT51, it should be greater than 0.25 for TiO_2 R104. The reason is that the threshold shear rate is slightly dependent on the inter-particle cohesion; it increases if the powder cohesiveness increases (Pasha et al., 2015). Note that, due to the particles size of the tested materials and the weight of the indenters, the hardness has not been directly investigated in the dynamic regime of flow.

Because the indentation results corroborate well with that reported by Tardos et al. (2003), the Ball Indentation Method represents a suitable technique that could be successfully used to characterise the flow resistance of powder beds. The main advantage is that it allows investigating shear rates that cannot be achieved with the common techniques. Nevertheless, the method exhibits some limitations. The first concerns the variability with the materials; e.g. for some powder a range of penetration depths with constant hardness cannot be determined. Secondly, it directly measures hardness, and therefore constraint factor must be determined to infer unconfined yield strength. Thirdly, the indenter diameter and drop height must be carefully selected to attempt to ensure the indenter penetration does not exceed the stable region, however since hardness may increase with strain rate by some unknown extent, this region cannot be guaranteed before testing. Finally, only powders with a certain degree of cohesiveness can be subjected to penetration.

References

Hassanpour A., Ghadiri M., 2007, Characterisation of Flowability of Loosely Compacted Cohesive Powders by Indentation, Particle & Particle Systems Characterization, 24, 117-123.

Hare C., Zafar U., Ghadiri M., Freeman T., Clayton J., Murtagh M. J., 2015, Analysis of the dynamics of the FT4 powder rheometer, Powder Technology, 285, 123-127.

Jenike A. W., 1964, Storage and flow of solids. Bulletin No. 123, Utah State University.

Klausner J. F., Chen D., Mei R., 2000, Experimental investigation of cohesive powder rheology, Powder Technology, 112, 94–101.

Leturia M., Benali M., Lagarde S., Ronga I., Saleh K., 2014, Characterization of flow properties of cohesive powders: A comparative study of traditional and new testing methods, Powder Technology, 253, 406-423.

Pasha M., Hare C., Hassanpour A., Ghadiri M., 2013, Analysis of ball indentation on cohesive powder beds using distinct element modelling, Powder Technology, 233, 80-90.

Pasha M., Hare C., Hassanpour A., Ghadiri M., 2015, Numerical analysis of strain rate sensitivity in ball indentation on cohesive powder Beds, Chemical Engineering Science, 123, 92-98.

Salehi H., Barletta D., Poletto M., Schütz D., Romirer R., 2017, On the use of a powder rheometer to characterize the powder flowability at low consolidation with torque resistances, AIChE Journal, 63, 4788-4798.

Santomaso A., Lazzaro P., Canu P., 2003, Powder flowability and density ratios: the impact of granules packing, Chemical Engineering Science, 58 (13), 2857-2874.

Schulze D., 2008, Powder and bulk solids. Behaviour, characterization, storage and flow. Springer, 35-74.

Tardos G. I., McNamara S., Talu I., 2003, Slow and intermediate flow of a frictional bulk powder in the Couette geometry, Powder Technology, 131, 23-39.

Tirupataiah Y., Sundararajan G., 1990, The volume of the crater formed by the impact of a ball against flat target materials-the effect of ball hardness and density, International Journal of Impact Engineering, 9, 237-246.

Zafar U., Hare C., Hassanpour A., Ghadiri M., 2017, Ball indentation on powder beds for assessing powder flowability: Analysis of operation window, Powder Technology, 310, 300-306.