

An automated apparatus for measuring the tensile strength and compressibility of fine cohesive powders

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This paper describes an apparatus based on a novel use of a powder bed, whereby the relationship between consolidation stress, tensile strength, and free volume of fine powder is measured. The powder to be tested is first initialized to a reproducible state. The initialized powder is next consolidated either beyond its own weight or below its own weight by means of a controlled flow of gas. An ultrasonic device measures the height of the bed, thus providing an average value of the powder free volume. Next the consolidated bed of powder is subjected to a slowly increasing gas flow, so directed as to put the powder under tension. The overpressure causing the powder to break provides a measure of the tensile strength of the powder, which in turn is a function of the consolidation and free volume. The relationship between consolidation stress, tensile strength, and free volume is related to powder flowability. © 2000 American Institute of Physics. [S0034-6748(00)03307-4]

I. INTRODUCTION

There is a need for an apparatus and technique for measuring the cohesive properties of fine cohesive powders such as xerographic toners, whose flow properties are of technological importance. Such flow properties are known to be closely related to interparticle forces. Flowability refers to the ease with which the bulk powder flows. A direct way of testing powder flowability is to make a standard amount of powder flow through a specified device.¹ For example, one test consists of measuring the time for the powder to be discharged from a hopper. A commercially available powder flowability tester,² frequently used for pharmaceutical tabletting applications, is based on the ability of the sample to flow freely through a hole in a disk. The diameter of the smallest hole through which the sample passes three consecutive times is taken as the flowability index. Another suggested property to characterize the flowability of the material is its angle of repose.³ However, as is well known, this property is not well defined for cohesive powders since it depends on the height of the pile (cohesive materials sometimes exhibit slopes steeper than 90°).

A common tool used to characterize the flowability of a material is the Hosokawa Powder Tester, which is based on the work of Carr.⁴ This test involves placing the powder on a series of screens and vibrating the screens vertically for a fixed period of time. The amount of powder remaining on each screen is measured and then used to calculate a flowability index. Some problems of these measurements include the large variability of the results between operators and even between measurements made by the same operator.

This problem is related to the absence of a reproducible initial state of the sample as the behavior of cohesive powders is very sensitive to the previous history. In addition, the calculated number is somewhat arbitrary depending on some external factors: size and material of the screens, vibration frequency, which does not always make the number consistent across different groups of people. In general, the results are not only dependent on the physical properties of the powder (particle size and shape, density, porosity, cohesivity, etc.) but also on the design and characteristics of the device.

To avoid these problems, there is need for a more consistent and more meaningful method of characterizing the powder flow. This method should be based on the direct measurement of the physical properties relevant to powder flow, like the tensile strength and free volume as a function of the consolidation stress. There are some commercially available instruments to test the tensile stress of powders like the split cell tester^{5,6} and the lifting-lid tester.⁵ However, for fine cohesive powders they all have in common the problem of how to achieve a uniform state of consolidation.

The average particle size is related to the powder flowability but we may have powders with the same particle size and different cohesivities, because of the use of flow additives that change mechanical surface properties like contact hardness and adhesion energy.^{7,8} The way a powder packs is directly related to its flow behavior in the sense that powders that pack well generally tend to flow well. The Hausner ratio,⁹ for example, is defined as the ratio of the tap density to the aerated bulk density and is used in many aspects of general powder technology. The compressibility of the powder is a useful measure of the powder flow characteristics and therefore the behavior of powders in compres-

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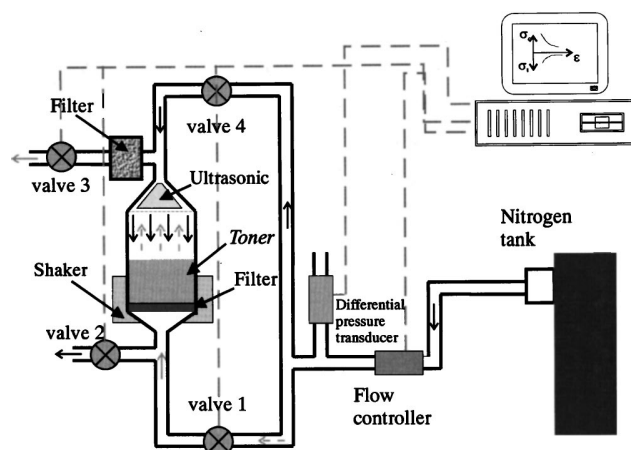


FIG. 1. Scheme of the apparatus. The powder sample is held in a 4.45-cm-diam, 17 cm height cylinder made of polycarbonate, the base of which is a sintered metal filter of $5\ \mu\text{m}$ pore size. The cylinder is placed on a shaker used to help powder fluidization. Dry nitrogen is supplied from a tank of compressed gas and a mass flow controller is used to adjust the flow. The pressure drop across the bed is measured by a differential pressure transducer. A series of computer controlled valves are used to control the gas flow path through the bed. The bed height is read from an ultrasonic sensor. All these components are connected to a computer by means of a data acquisition board. Another porous filter is placed before valve 3 to catch elutriated particles and prevent damage to valve 3.

sion can be used as a technique to measure powder flowability.

Poor flowing materials pack in a very open structure, and are very compressible, whereas materials that flow well tend to pack closely and are not so compressible. Clearly, therefore, it would be desirable to measure compressibility and its relation to packing structure in order to characterize flowability. Compaction tests are based on this concept, and there exists a method in which a cylindrical plug of the granular material is compressed axially by a piston which generates pressures ranging from 10 to 3200 kPa; this method is used in testing the consolidation and flow properties of soils for civil engineering purposes.¹⁰

From our experience in handling xerographic toners,^{8,11} their flow behavior is sensitive to consolidation at very low stresses (in the range from about 1 to a few hundred Pa). Thus a technique is needed in order to subject a powder to very small consolidation stresses, to measure the free volume of the powder and its tensile strength as a function of consolidation stress. The apparatus described in this paper was designed to perform this type of measurement in a computer-controlled, automated process.¹² Apart from characterizing the flow of materials this apparatus can be used as a research tool to gain a better understanding about powder flow and the contact forces between particles.^{7,11}

II. DESCRIPTION OF THE APPARATUS AND PROCEDURE: EXPERIMENTAL RESULTS

A schematic view of the apparatus is shown in Fig. 1. The powder bed is contained in a cylinder made of polycarbonate, 2.54 cm inner diameter and 17 cm in height with a porous, sintered metal filter base ($\sim 5\ \mu\text{m}$ pore size). The powder can be subjected to a controlled gas flow either up-

wards or downwards. Dry nitrogen, dispensed from a tank of compressed gas, is used in order to avoid problems associated with an increase in cohesion due to humidity. The flow path is controlled by the computer which actuates the solenoid valves shown in Fig. 1. When valves 1 and 3 are open and 2 and 4 are closed the flow is directed upward through the powder bed. When valves 2 and 4 are open and 1 and 3 are closed the flow is directed downward. The gas flow is controlled by a MKS mass flow controller in the range from zero to $200\ \text{cm}^3/\text{min}$. An analog signal generated by the computer sets the flow to the desired value. The gas pressure drop across the powder is measured by a MKS differential pressure transducer that generates an analog signal that can be read by the computer. Note that the pressure drop measured has two components, the pressure drop across the powder Δp and the pressure drop across the filter Δp_f . A preliminary calibration procedure measures the filter pressure drop. This calibration consists of measuring the resistance of the porous plate without the powder. Then Δp is obtained by subtracting Δp_f from the total pressure drop. The sample holder is mounted on a vibrator which is used to help in the fluidization of very cohesive powders. A solid-state relay that can be operated from the computer controls the activation of the vibrator. The height of the powder bed h is measured by means of an acoustic pulse technique. To this end an ultrasonic sensor is located at the top of the sample holder, and the analog output provided by the sensor is read and translated by the computer to h through a proper calibration. Then, from the mass of the sample and the density of the polymer, the value of h enables us to calculate the average free volume of the powder. Finally a low resistance filter is placed in the line as indicated in Fig. 1 to trap the particles elutriated by the gas and prevent the contamination of valve 3. All the components of the powder tester are controlled and coordinated from the computer according to a proper operating procedure to measure the relationship between consolidation stress, tensile strength, and free volume of the powder.⁷

Using this technique we have tested a series of model xerographic toners designed for us at Xerox Corporation. These toners are based on one batch of styrene-butadiene powder, particle diameter $12.7\ \mu\text{m}$ (volume average), with varying weight concentration of the fumed silica flow control additive Aerosil® (tradename of Degussa Corporation). These toners are designated RT-5462 (0.01% additive); RT-5114 (0.05% additive); RT-5115 (0.2% additive). In addition a commercially available toner, Canon CLC 500 ($8.5\ \mu\text{m}$ particle diameter) has also been tested. A detailed description of the flow properties of these materials is given elsewhere.⁷

First step: Fine cohesive powders tend to pack in a very heterogeneous manner with some regions close packed and other regions very open in structure; clearly such powders are difficult to characterize. Therefore our first step is to initialize the powder to a reproducible state of consolidation. A controlled gas flow is blown through the bed and above a critical gas flow velocity the bed becomes fluidized. In the case of highly cohesive powders it is necessary to vibrate the bed while fluidizing to break up channels and bubbles which would otherwise prevent fluidization. When it is fluidized the

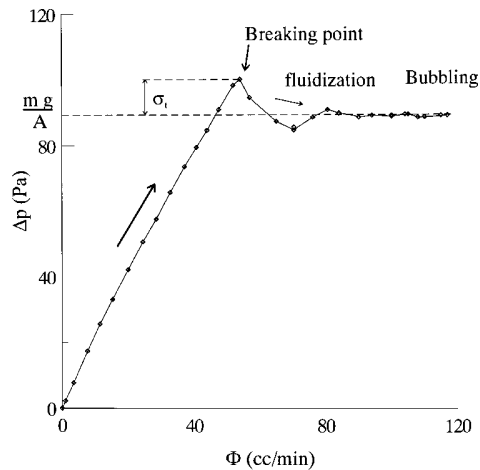


FIG. 2. Pressure drop across the powder bed vs gas flow for a xerographic toner (RT 0.1 wt % of silica, mass 29 g, filter area 25 cm²) in the breaking step. The pressure drop and, hence the drag force exerted on the particles, increases linearly with the flow rate, while the bed remains static. The point at which the pressure drop equals the weight of the powder is the point of minimum fluidization gas flow. If the gas flow is further increased the pressure drop continues to increase until interparticle forces are overcome, at which point a sudden decrease in the pressure drop is observed. The overshoot of the pressure drop beyond the weight of the bed gives the value of the tensile strength of the powder.

powder is uniform, so that it loses all memory of its initial, heterogeneous state. If the gas flow is turned off, the fluidized powder settles into a reproducible state. In the settled state the powder is not perfectly uniform as the bottom of the bed is somewhat more consolidated than the powder at the top, but it is the compressed powder at the bottom that is of concern in these measurements; the consolidation stress σ_c at the bottom of the bed is given by the weight per unit area of the powder,

$$\sigma_c = \frac{mg}{A}, \tag{1}$$

where m is the mass of powder and A is the area of the bed.

Second step: The average void fraction ϵ for a given degree of consolidation is obtained by measurement of the bed height h , given by the ultrasonic transducer,

$$\epsilon = 1 - \frac{m}{\rho_p Ah}, \tag{2}$$

where ρ_p is the particle density, which we measure via an Accu Pyc 1330 Pycnometer.

Third step: The powder layer is subjected to a slowly increasing gas flow directed upwards through the bed. At first the bed structure is unperturbed and the pressure drop increases linearly (see Fig. 2). This line corresponds to Carman's law¹³ and the greater the void fraction the smaller the slope. At the point of minimum fluidization velocity the pressure drop across the powder layer balances the weight of the powder per unit area, and at this point a powder with zero cohesion will become fluidized. In practice, all powders exhibit some degree of cohesivity, and so the pressure drop across the bed continues to increase above the minimum fluidization velocity point. Note that above this point the gas flow puts the bed under tension, and as the tension builds up

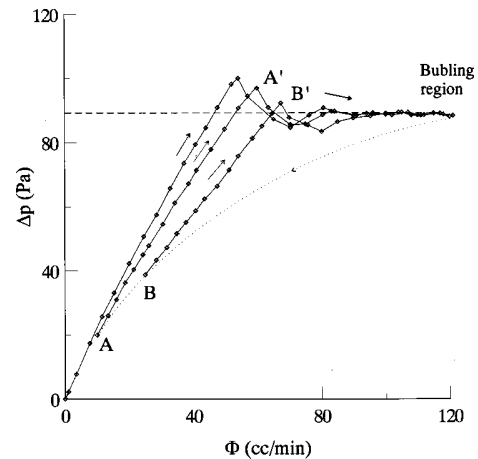


FIG. 3. Pressure drop across the powder bed vs gas flow for a xerographic toner (RT 0.1 wt % of silica, mass 29 g, filter area 25 cm²) in the breaking step. A and B initial states correspond to underweight consolidated states. The slope of the linear part decreases as the consolidation stress of the initial state decreases indicating a larger free volume of the sample. The tensile strength, given by the overshoot of the pressure drop beyond the weight of the bed (points A' and B'), also decreases as the consolidation stress is reduced.

there comes a point at which the powder breaks in tension (see Fig. 2). We observe that the fracture of the bed always starts at its lowest point, the bottom of the bed. Therefore, the condition for tensile yield will be met first at the contact between the bed and the filter or within the bed at the bottom, not a higher level. In all cases we have studied we observed that the filter surface remains covered by a thin layer of powder and therefore we conclude that the condition for tensile yield stress is that of the powder at the bottom of the bed, and not the powder-filter interface.⁷ The tensile strength σ_t of the powder is given by the difference between the pressure drop (which is continuously measured) across the bed just before the breaking and the weight per unit area,

$$\sigma_t = (\Delta p)_{\max} - \frac{mg}{A}. \tag{3}$$

Fourth step: From the process described above, we obtain¹¹ the tensile strength and average free volume of the powder for the consolidation stress given by its weight per unit area. To change the consolidation stress we may add or subtract powder to the bed,⁷ an alternative technique is to consolidate the powder layer in a centrifuge.¹⁴ We have used both of these processes but they are tedious and cannot be automated. The idea behind this new method is to keep the mass constant and to change the consolidation by means of direct or reverse gas flow.

A. Underweight consolidation

The gas flow is increased up to the fluidized region where the powder's memory of the previous cycle is erased, then the gas flow is decreased to a nonzero value and the powder is allowed to settle down under the remaining flow. At that point (point A in Fig. 3) the consolidation stress is given by

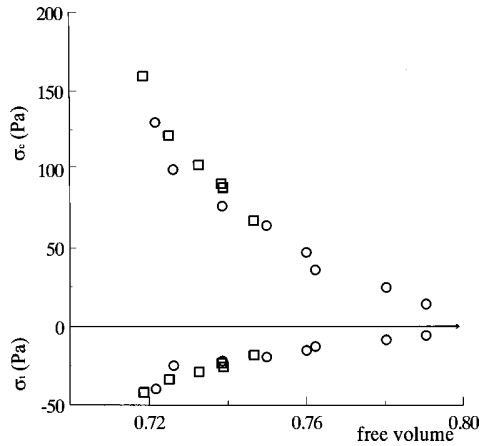


FIG. 4. Measured relationship between consolidation stress, tensile strength, and free volume for a xerographic toner (RT 0.01 wt % of silica). ○: Varying the consolidation stress changing the mass of powder. □: Reducing the consolidation by means of a forward gas flow. The size of error bars is of the order of symbols size.

$$(\sigma_c)_A = \frac{mg}{A} - (\Delta p)_A \quad (4)$$

The ultrasonic transducer measurement of the bed height then gives the free volume of the sample under this new consolidation stress. Then the gas flow is again slowly increased until the powder breaks (point A' in Fig. 3) enabling us to measure the new tensile strength. Notice that the slope of the linear part is now smaller, indicating a larger free volume. Another observation is that, as expected, the tensile strength decreases as the consolidation stress is reduced. The process of fluidization and underweight consolidation is then repeated, many times, and in this way we have been able to measure ϵ and σ_t down to very small consolidation stresses as low as 10 Pa. Stresses are measured within a precision of ± 3 Pa. In Fig. 4 an example is shown of the relationship between consolidation stress, tensile strength, and free volume of a cohesive powder (xerographic toner RT 5462) measured by this process of underweight consolidation. In Fig. 5

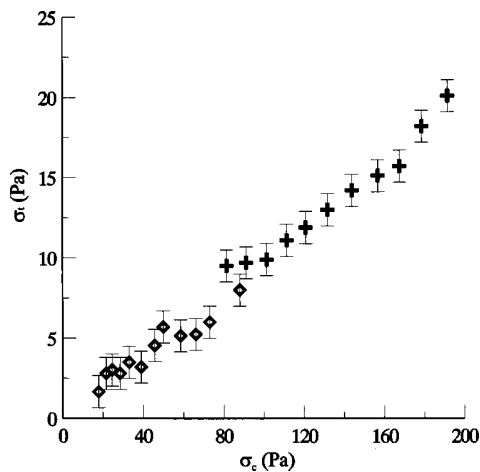


FIG. 5. Tensile strength vs consolidation stress for a xerographic toner (Canon CLC500). +: Varying consolidation stress by changing the mass of toner. ◇: Reducing the consolidation by means of a gas flow from the same initial mass of toner.

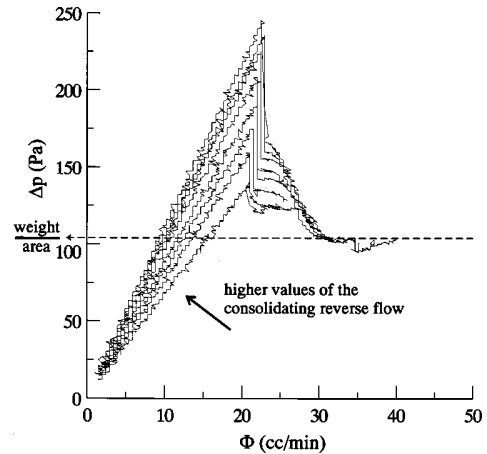


FIG. 6. Gas pressure drop vs gas flow for the same mass of a xerographic toner (RT 0.05 wt % of silica) after being consolidated with increasing values of a reverse gas flow.

we see that the tensile strength of this cohesive powder increases linearly with consolidation stress in the range of low consolidation stresses. In Figs. 4 and 5 we also included experimental points from our earlier work⁷ obtained by changing the consolidation stress through changes in the mass to the sample. As can be seen, the results from the two techniques agree within the experimental scatter.

B. Overweight consolidation

In order to create consolidation stresses above the weight of the sample we again use the gas flow to achieve this purpose. After the initialization step, when the bed is settled under its own weight the flow path is reversed and the gas flow is slowly increased to the desired value. The consolida-

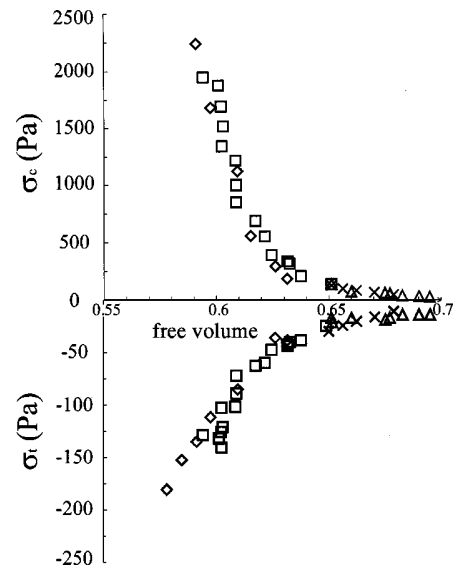


FIG. 7. Relationship between consolidation stress, tensile strength, and free volume for a xerographic toner (RT 0.2 wt % of silica) using different techniques of consolidation of the sample. □: Increasing the consolidation by means of a reverse gas flow. △ and ×: Reducing the consolidation by means of a forward gas flow. ◇: Increasing consolidation by centrifuging the sample. Please note that the positive and negative axes are in different scales.

tion stress at the bottom of the bed is then given by adding the gas pressure drop across the layer to the weight per unit area of the initialized powder,

$$\sigma_c = \frac{mg}{A} + \Delta p_A. \quad (5)$$

Once the bed is compressed, the gas flow is reduced to zero and the flow path is again reversed to measure the tensile strength of the powder, in the way described previously. From Fig. 6 it is clear that as the consolidation increases the tensile strength increases. Also the slopes of the pressure drop versus flow velocity shown in Fig. 6 are higher as the sample is more consolidated, indicating a decrease in the average free volume, as expected. In Fig. 7 we show the relationship between consolidation stress, tensile strength, and free volume of the xerographic toner RT 5114 (0.05% additive). An alternative technique introduced for comparison is that of consolidation by centrifuging the sample. As can be seen all data fall in the same curve within experimental scatter, independent of the consolidation technique used.

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