INTERNATIONAL

Designation: D7891 – 15

Standard Test Method for Shear Testing of Powders Using the Freeman Technology FT4 Powder Rheometer Shear Cell[1]

This standard is issued under the fixed designation D7891; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This method covers the apparatus and procedures for measuring the incipient failure properties of a powder as a function of the normal stress for a given level of consolidation. The method also allows the further determination of the unconfined yield strength, internal friction angles, cohesion, flow function, major principal stress and wall friction angle (with the appropriate wall coupon fitted to the correct accessory).

1.2 These parameters are most commonly used for the design of storage hoppers and bins using industry standard calculations and procedures. They can also provide relative classification or comparison of the flow behavior of different powders or different batches of the same powder if similar stress and shear regimes are encountered within the processing equipment.

1.3 The apparatus is suitable for measuring the properties of powders with a maximum particle size of 1 mm. It is possible to test powders which have a small proportion of particles of 1 mm or greater, but they should be present in the bulk sample as no more than 5 % of the total mass in samples with a normal (Gaussian) size distribution.

1.4 All observed and calculated values shall conform to the guidelines for significant digits and rounding established in Practice D6026.

1.4.1 The procedures used to specify how data are collected/recorded or calculated, in this standard are regarded as the industry standard. In addition, they are representative of the significant digits that generally should be retained. The procedures used do not consider material variation, purpose for obtaining the data, special purpose studies, or any considerations for the user's objectives; and it is common practice to increase or reduce significant digits of reported data to be commensurate with these considerations. It is beyond the scope of this standard to consider significant digits used in analysis methods for engineering design.

1.5 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D653 Terminology Relating to Soil, Rock, and Contained Fluids

D2216 Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass

D3740 Practice for Minimum Requirements for Agencies Engaged in Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction

D6026 Practice for Using Significant Digits in Geotechnical Data

D6128 Test Method for Shear Testing of Bulk Solids Using the Jenike Shear Cell

D6682 Test Method for Measuring Shear Stresses of Powders Using Peschl Rotational Split Level Shear Tester

D6773 Test Method for Bulk Solids Using Schulze Ring Shear Tester

3. Terminology

3.1 *Definitions*—For definitions of common technical terms in this standard, refer to Terminology D653.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *conditioning, v*—in powders, the process of homogenizing the stress of a powder specimen by use of a specialized blade attachment.

3.2.2 *wall friction coupon, n*—in powders, a test piece used in the wall friction test that is manufactured from a material that represents the material of construction of the silo/bin/hopper that stores the powder.

4. Summary of Test Method

4.1 *Selection of the Appropriate Testing Regime*—The particular consolidating stress level or levels used to evaluate the

[1] This test method is under the jurisdiction of ASTM Committee D18 on Soil and Rock and is the direct responsibility of Subcommittee D18.24 on Characterization and Handling of Powders and Bulk Solids.

Current edition approved March 1, 2015. Published March 2015. DOI: 10.1520/D7891-15.

flow properties of the powder will depend on the reason for generating the data, as outlined in Section 5, and should broadly reflect the stresses that the powder will be subjected to in its processing environment.

4.2 *Preparation of the specimen*—The specimen is added to the test vessel and its mass determined using the instrument's built-in balance. The selected test program is then initiated and runs independently of the operator other than the interchange of the spindle-mounted attachments for different sections of the test. The powder first undergoes a conditioning cycle using the blade attachment which removes any variability introduced during filling or from the material's previous history. The piston attachment is then fitted and is used to compress the powder to the required consolidating stress as determined in the selected test program. Excess powder is then removed from the test cell by means of a leveling assembly to leave a specimen of compressed powder with a level surface that is ready for shear testing. The shear head is then fitted to the instrument.

4.3 *Measurement of Shear Stress*—The instantaneous shear stress is then measured by re-establishing the consolidating stress with the shear head and then pre-shearing the specimen until a steady state condition is reached. The powder is then subjected to a reduced normal load and then sheared until the shear force reaches a maximum and then decreases.

4.4 *Measurement of Wall Friction as a Function of Normal Stress*—The same specimen preparation method is used for this test, but a wall friction attachment, fitted with a coupon representing the material against which the powder is required to flow, is used instead of a shear head.

5. Significance and Use

5.1 The test can be used to evaluate the following:

5.1.1 *Classification or Comparison of Powders*—There are several parameters that can be used to classify powders relative to each other, the most useful being the measured shear stresses, cohesion, flow function and angle of internal friction.

5.1.2 *Sensitivity Analysis*—The shear cell can be used to evaluate the relative effects of a range of powder properties and/or environmental parameters such as (but not limited to) humidity, particle size and size distribution, particle shape and shape distribution, moisture content and temperature.

5.1.3 *Quality Control*—The test can, in some circumstances, be used to assess the flow properties of a raw material, intermediate or product against pre-determined acceptance criteria.

5.1.4 *Storage Vessel Design*—Mathematical models exist for the determination of storage vessel design parameters which are based on the flow properties of powders as generated by shear cell testing, requiring shear testing at a range of consolidating stresses as well as the measurement of the wall friction angle with respect to the material of construction of the storage vessel. The methods are detailed in Refs. (**1-3**).[2]

NOTE 1—The quality of the result produced by this test method is dependent on the competence of the personnel performing it, and the suitability of the equipment and facilities used. Agencies that meet the criteria of Practice D3740 are generally considered capable of competent and objective testing/sampling/inspection/etc. Users of this test method are cautioned that compliance with Practice D3740 does not in itself assure reliable results. Reliable results depend on many factors; Practice D3740 provides a means of evaluating some of those factors (**4**).

Practice D3740 was developed for agencies engaged in the testing and/or inspection of soil and rock. As such it is not totally applicable to agencies performing this test method. However, users of this test method should recognize that the framework of Practice D3740 is appropriate for evaluating the quality of an agency performing this practice. Currently there is no known qualifying national authority that inspects agencies that perform this test method.

6. Apparatus

6.1 The FT4 Powder Rheometer is shown in Fig. 1. It is a computer-controlled instrument which simultaneously measures the force and torque required to mobilize a powder contained in a range of vessel types using a series of spindle-mounted attachments driven by an electric motor located on a carriage, driven by another electric motor, which moves the attachments in the vertical direction.

6.1.1 The force is measured by a force transducer located beneath and fixed to the table that supports the test vessel during the measurement process.

6.1.2 The torque (shear resistance) is evaluated by measuring the moment on the attachment using a torque transducer.

6.2 The shear cell vessel is shown in Fig. 2. It consists of a serrated base, made from a suitable engineering plastic such as polyoxymethylene (POM), onto which are mounted two borosilicate glass cylinders (50-mm × 85-mL vessel) connected by a POM leveling assembly.

6.2.1 The shear cell vessel is located on the powder rheometer using a POM clamp ring.

6.2.2 A POM funnel is also fitted to assist with the filling of the vessel.

6.3 Attachments are fitted to the powder rheometer to facilitate various test procedures.

6.3.1 The first is a twisted blade (shown in Fig. 3(A)) that is used to condition the test specimen thus generating a repeatable stress condition within the powder.

NOTE 2—This conditioning process eliminates the effects of the powder's history and also any operator-induced effects generated during the filling process.

6.3.2 The second (Fig. 3(B)) is a compaction piston that compresses the specimen to achieve the desired consolidating normal stress.

6.3.3 The third (Fig. 3(C)) is a shear head consisting of 18 blades that are used to generate shearing within the powder.

6.3.4 The fourth (Fig. 3(D)) is a wall friction head and an interchangeable coupon representing the material of construction against which the powder will be required to flow.

6.3.5 All of the attachments (Fig. 3) are made from stainless steel and stainless steel+anodized aluminum.

NOTE 3—The blades located in the shear head are thin and thus relatively sharp. Care must be taken when handling the shear head to prevent skin abrasions and cuts.

6.4 Additionally, it is possible to employ shear cells with 10 mL and 1 mL capacity in conjunction with the FT4 Powder

[2] The boldface numbers in parentheses refer to a list of references at the end of this standard.

FIG. 1 FT4 Powder Rheometer (The left hand image shows the instrument with the shear head fitted; the right hand image shows the shear head and shear cell vessel.)

FIG. 2 Shear Cell Vessel

Rheometer if the quantity of available test specimen is less than 85 mL (at the chosen consolidation stress). The mode of operation of the 10-mL shear cell is identical to that described herein for the 85-mL shear cell but using a smaller shear cell and range of attachments. The limit on the maximum particle size is commensurately reduced to a maximum particle size of 0.5 mm. The 1-mL shear cell uses a significantly different cell design and attachments, which is beyond the scope of this standard.

7. Preparation of Apparatus

7.1 Since the integrity of the blades within the shear cell head is critical to generating accurate and reliable data, handle the shear head with care, store it in the case provided and inspect it for damage at regular intervals.

7.2 Make sure that the shear cell vessel components and the spindle-mounted attachments are clean and free from grease and other contaminants (**5**).

7.3 The following items are required to assemble the shear cell vessel: two 50-mm × 85-mL glass cylinders, a 50-mm serrated base; a 50-mm clamp ring; a 50-mm leveling assembly; and a 50-mm funnel. These items are shown in Fig. 4. A fully detailed assembly procedure is also available (**6**).

7.4 To assemble the shear cell vessel, position the clamp ring approximately 1 mm from the end of one of the glass cylinders and loosely fit the clamp ring onto the glass cylinder (Fig. 5). The clamp ring must not project past the end of the glass cylinder, otherwise misalignment may occur. Make sure that the gap in the clamp ring is approximately centralized with the printing on the glass cylinder. Secure the clamp ring using the hex driver ensuring that the screw is not over tightened.

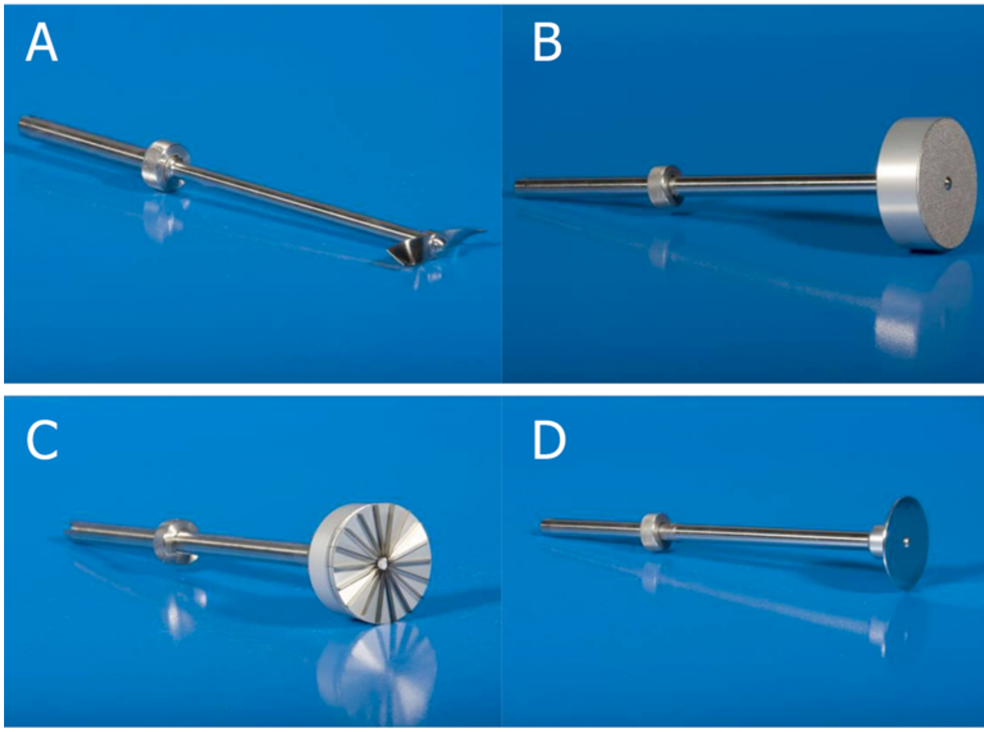

FIG. 3 Spindle-Mounted Attachments Used in Shear and Wall Friction Tests: Blade (A); Vented Piston (B); Shear Head (C); Wall Friction Head (D)

FIG. 4 Components Required to Assemble the 50-mm × 85-mL Shear Cell Vessel

7.5 Locate the serrated base into the glass cylinder adjacent to the clamp ring. Carefully rotate the serrated base to make sure that the entire circumference is in contact with the glass cylinder (Fig. 6).

7.6 Open the leveling assembly and place it on top of the glass cylinder at the opposite end to the clamp ring and serrated base. Make sure that the gap in leveling assembly is approximately in line with the gap in the clamp ring.

FIG. 5 Fitting the Clamp Ring

7.7 Carefully invert the glass cylinder, clamp ring, serrated base and leveling assembly and place on the edge of a flat surface (Fig. 7) so that the glass cylinder can be fitted flush with the inner face of the leveling assembly without impediment from the upper part of the leveling assembly.

7.8 Push down gently on both the glass cylinder and the leveling assembly so that they are both flush with the flat surface.

7.9 Tighten the leveling assembly with the hex driver such that the leveling assembly and the glass cylinder are securely located.

7.10 Confirm that the glass cylinder and leveling assembly are flush, and check that the leveling assembly operates smoothly.

7.11 Close the leveling assembly.

7.12 Place the other 50-mm × 85-mL glass cylinder into the top half of the leveling assembly and gently rotate the upper glass cylinder to make sure that it is in contact with the glass cylinder below.

7.13 Tighten the leveling assembly with the hex driver such that the leveling assembly and the upper glass cylinder are securely located (Fig. 8).

7.14 Place the funnel on top of assembled shear cell vessel (Fig. 9) and locate on the FT4 Powder Rheometer.

NOTE 4—The assembled shear cell vessel is described as a 50-mm × 85-mL split vessel assembly, which indicates the glass cylinders' internal diameter and the precise volume of the lower section of vessel with the base fitted.

8. Calibration and Standardization

8.1 Calibrate the instrument in accordance with the manufacturer's instructions.

NOTE 5—The force and torque transducers located within the instrument are calibrated using proprietary fixtures in conjunction with calibration masses that are supplied with the instrument. (**7**)

9. Procedure

9.1 With the assembled shear cell vessel (Section 7) located on the instrument table, tare (zero) the mass of the empty shear cell vessel using the built-in balance prior to filling with the test specimen.

9.2 Once tared, remove the shear cell vessel for filling.

9.3 Fill the tared shear cell vessel with sufficient powder such that, following the compression stage, the specimen is not compressed below the split level of the leveling assembly.

NOTE 6—The amount of specimen required depends on the compressibility of the particular powder and the consolidating stress level chosen for the test. If the powder's compressibility with respect to the consolidating stress is known from a previously completed compressibility test (**8**), the required mass of uncompressed powder can be determined based on the chosen consolidating stress of the shear test.

NOTE 7—If the level of the powder is below the level of the leveling assembly following the compression phase, the test should be classified as a failure and re-run with a greater starting volume.

9.4 Return the filled shear cell vessel and securely fasten it to the instrument table using the clamping assembly. The mass of the powder specimen is then registered within the data file associated with the test.

9.5 *Measurement of Shear Stress:*

9.5.1 Select the appropriate test program from the program library. There are four standard test programs available which are based on consolidating stresses of 3, 6, 9 and 15 kPa. These programs can be modified if other consolidating stresses are required.

NOTE 8—For advanced users the test method can be modified in detail with respect to shear rate, number and length of pre-shear cycles. These options are detailed in Annex A1 and Ref. (**9**).

9.5.2 Once selected, the test program will display images of the correct test vessel and attachment that are required to commence the test. For a shear test, the shear cell vessel and blade are used for initiating the preparation of the sample.

9.5.3 Push the start button on the computer screen to commence the test program which will cause the blade to be slowly lowered into the shear cell vessel after which it will perform a conditioning cycle by traversing through the powder along a prescribed helical path.

9.5.4 Once the conditioning cycle is complete and the test has been automatically paused, exchange the blade attachment for the vented piston attachment following the on-screen instructions.

9.5.4.1 After the exchange has been confirmed, the program will continue by moving the vented piston attachment into the shear cell vessel to compress the powder until the target consolidating stress has been achieved and held for 60 seconds.

9.5.5 Once the compression cycle is complete and the test has been automatically paused, remove the funnel.

9.5.6 After confirming this step, use the leveling assembly to separate and remove the excess powder left in the upper section of the shear cell vessel, and collect it in a suitable container.

9.5.7 Leaving the leveling assembly in the open position, replace the vented piston with the shear head.

FIG. 6 Fitting the Serrated Base

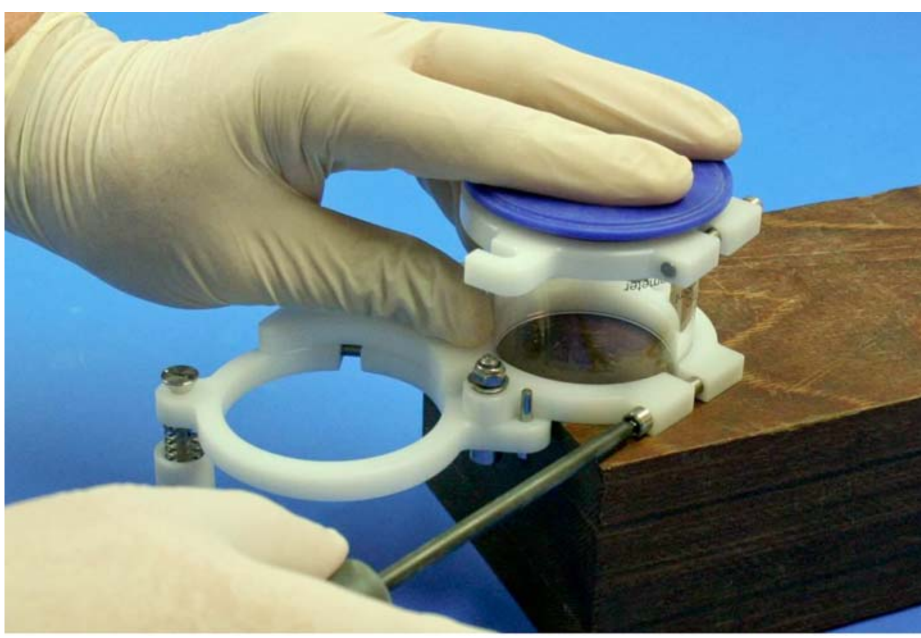

FIG. 7 Fitting the Leveling Assembly

9.5.8 Move the shear head to the surface of the powder at a speed of 0.5 mm/s and then slowly move it down at a maximum speed of 0.08 mm/second until the target consolidating stress is re-established and held for 60 seconds.

9.5.9 Perform a pre-shearing cycle by rotating the shear head within the powder at a fixed rate of 18 degrees/min while maintaining the target consolidating stress until steady state shear stress has been achieved.

9.5.10 Stop rotating the shear head in the direction of shear and establish a zero torque level by rotating in the opposite direction.

9.5.11 Repeat 9.5.9 and 9.5.11 a maximum of ten times or until two consecutive steady state shear stress values are within 1 % of each other.

NOTE 9—If convergence does not occur in ten cycles, the powder is unstable, possibly due to degradation within the shear zone and may mean that the powder is not suitable for shear testing.

NOTE 10—The operator may modify the test programs to utilize alternative pre-shearing schemes if required (Annex A1).

9.5.12 Once the pre-shear requirements are met, initiate the shear testing cycle.

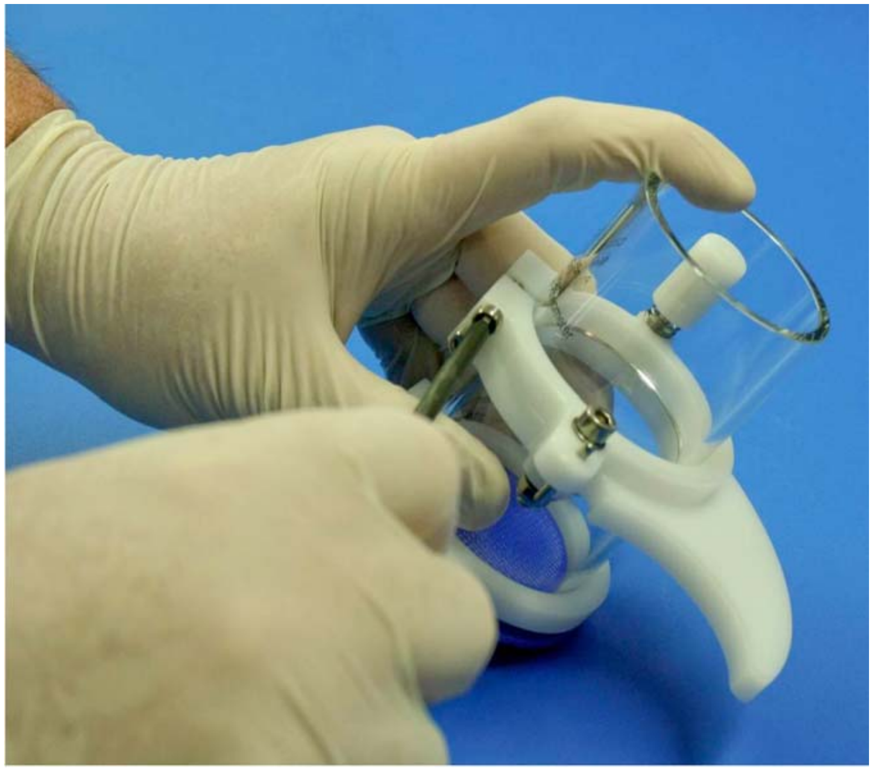

FIG. 8 Fitting the Upper Glass Cylinder

FIG. 9 Shear Cell Vessel Located on Instrument Table Prior to Taring (Zeroing) of the Empty Shear Cell Vessel Mass

9.5.12.1 Reduce the normal stress on the specimen to a pre-selected value lower than the consolidating stress (typically an integer value which is ~70-80 % of the consolidating stress) and hold for 20 seconds while maintaining zero torque.

9.5.12.2 Re-initiate shear until a maximum shear stress is achieved and the shear stress has subsequently reduced.

NOTE 11—The maximum shear stress recorded is the peak shear point for the selected normal stress.

9.5.13 Re-establish the consolidating stress and then re-initiate a single pre-shearing cycle.

9.5.14 Once steady state pre-shear has been re-established at the consolidating stress, use the program to select another lower normal test stress and repeat 9.5.13 and 9.5.14.

9.5.15 Repeat 9.5.12 to 9.5.15 until the program has completed all the test conditions that have been specified.

9.5.16 The standard test routines contain five measurement points between ~20-80 % of the consolidating stress, but the operator can modify the program to utilize additional or alternative measurement points if required.

9.6 *Measurement of Wall Friction:*

9.6.1 The measurement of wall friction follows a similar protocol to the measurement of the powders' shear stress except that, instead of using the shear head, an attachment is used, which has been fitted with a 48-mm diameter coupon of the material of construction to be evaluated.

NOTE 12—This material represents the wall of a storage vessel (or other powder processing equipment) against which the powder will be required to interact.

9.6.2 Make sure that the selected wall material coupon is clean, dry and free of any surface contaminants.

9.6.3 Repeat 9.1 to 9.5.6.

9.6.4 Once the compression cycle is complete and the instrument has paused the test program, remove the funnel.

9.6.5 Use the leveling assembly to separate and remove the excess powder left in the upper section of the shear cell vessel, which is collected in a suitable container.

9.6.6 Leaving the leveling assembly in the open position, replace the vented piston with the wall friction attachment.

9.6.7 Move the wall friction attachment to the surface of the powder, then move the head further down at a maximum speed of 0.08 mm/second until the target consolidating stress is re-established and held for 60 seconds.

9.6.8 Perform a pre-shearing cycle by rotating the wall friction attachment against the powder at a fixed rate of 18 degrees/min while maintaining the target consolidating stress until a steady state shear stress has been achieved.

9.6.9 Stop rotating the wall friction attachment in the direction of shear and establish a zero torque level by rotating in the opposite direction.

NOTE 13—The standard wall friction programs have a single pre-shear as a default setting.

9.6.10 Reduce the normal stress on the specimen to a pre-selected value lower than the consolidating stress and hold for 20 seconds while maintaining zero torque.

9.6.11 Re-initiate shear until a maximum shear stress is achieved and the shear stress has subsequently reduced.

9.6.12 Maintain shear for 45 seconds such that the kinematic shear stress can be calculated.

9.6.13 Stop rotating the wall friction attachment in the direction of shear and establish a zero torque level by rotating in the opposite direction.

9.6.14 Establish the next normal stress level and re-initiate rotation of the wall friction attachment.

9.6.15 Repeat 9.6.13 and 9.6.14 until all the test conditions have been completed.

9.6.16 The standard test routines contain five measurement points between ~20-80 % of the consolidating stress, but the operator can modify the program to utilize additional or alternative measurement points if required.

10. Calculation or Interpretation of Results

10.1 Record the force, torque and position values generated during the test program.

NOTE 14—This data, which is stored in a computer file, can be evaluated using the software program Data Analysis, which is provided with the Freeman Technology FT4 Powder Rheometer.

10.2 Calculate the normal stress by dividing the normal force measured by the instrument load cell by the cross-sectional area of the shear head.

$$\sigma = \frac{N}{A} \quad (1)$$

where:

σ = normal stress (kPa)
N = measured force (N)
A = area of the shear head (m^2)

10.3 Calculate the shear stress from the measured torque using the following equation:

$$\tau = \frac{3 \cdot T}{2 \cdot \pi \cdot r^3} \quad (2)$$

where:

τ = shear stress (kPa)
T = measured torque (Nm)
r = radius of the shear head (m)

10.4 Use pro-rating, if necessary, to compensate for slight changes in pre-shear stress during a series of shear measurements. Pro-rating involves recalculating the shear stress for each test point using the following formula:

$$\tau_{s,pro-rated} = \tau_s \times \frac{\tau_{p\ average}}{\tau_p} \quad (3)$$

where:

$\tau_{s,pro\text{-}rated}$ = pro-rated shear stress (kPa)
τ_s = measured shear stress (kPa)
$\tau_{p\ average}$ = average of all pre-shear shear stresses (kPa)
τ_p = measured pre-shear shear stress preceding shear step (kPa)

10.5 Plot the pre-shear point and all the valid shear test points as normal stress/shear stress data pairs (Fig. 10). Draw a least mean squares, linear regression line through the test points, and extrapolate this line to the normal stress level of the pre-shear point and, in the opposite direction, to meet the y-axis (where the normal stress is zero). This line is known as the yield locus. The angle that the yield locus makes with the horizontal axis in known as the angle of internal friction and is designated as φ. The point at which the yield locus crosses the y-axis is known as the cohesion, C.

10.6 Check to make sure that the yield locus passes above or through the pre-shear point. If it passes below the pre-shear point, scrutinize the individual test points to make sure that they are valid or re-run the test.

10.7 Draw a Mohr stress circle such that its center is located on the x-axis, it is tangential to the yield locus, and the origin is a point on the circle. The non-zero intersection of this circle with the x-axis defines the unconfined yield strength, f_c.

10.8 Draw a second Mohr stress circle such that its center is located on the x-axis, it passes through the pre-shear point, and it is tangent to the yield locus. This circle intersects the x-axis at two positions—the greater of which defines the major principal stress, σ_1, seen in the powder at this level of consolidation during steady state flow, as shown in Fig. 10.

10.9 Draw a line from the origin that is tangential to the major Mohr circle. This line is the effective yield locus, and the angle that this line subtends to the x-axis is the effective angle of friction, δ.

10.10 Construct a flow function, FF, by drawing a smooth curve through a series of data points, each of which represents a pair of values of major principal stress and unconfined yield strength.

10.11 *Calculation of Wall Friction Angle:*

10.11.1 Plot the pre-shear point and all valid shear test points as normal stress/shear stress data pairs (Fig. 11). Draw a least mean squares regression line through all the data points.

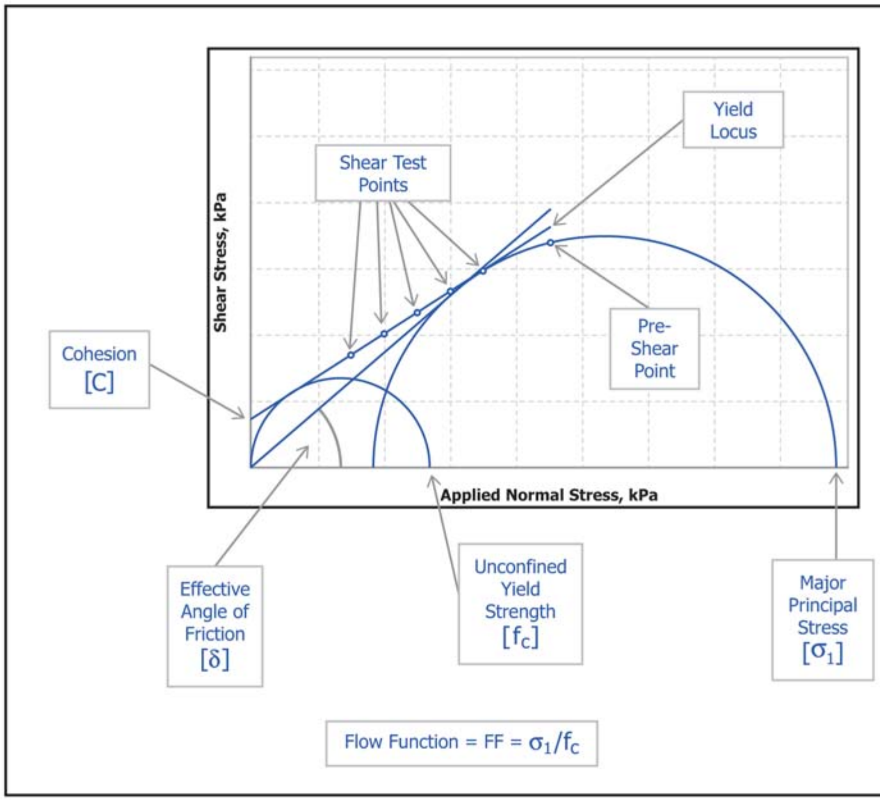

FIG. 10 Graphical Representation of the Shear Points, Mohr Circle Constructions and the Derived Parameters

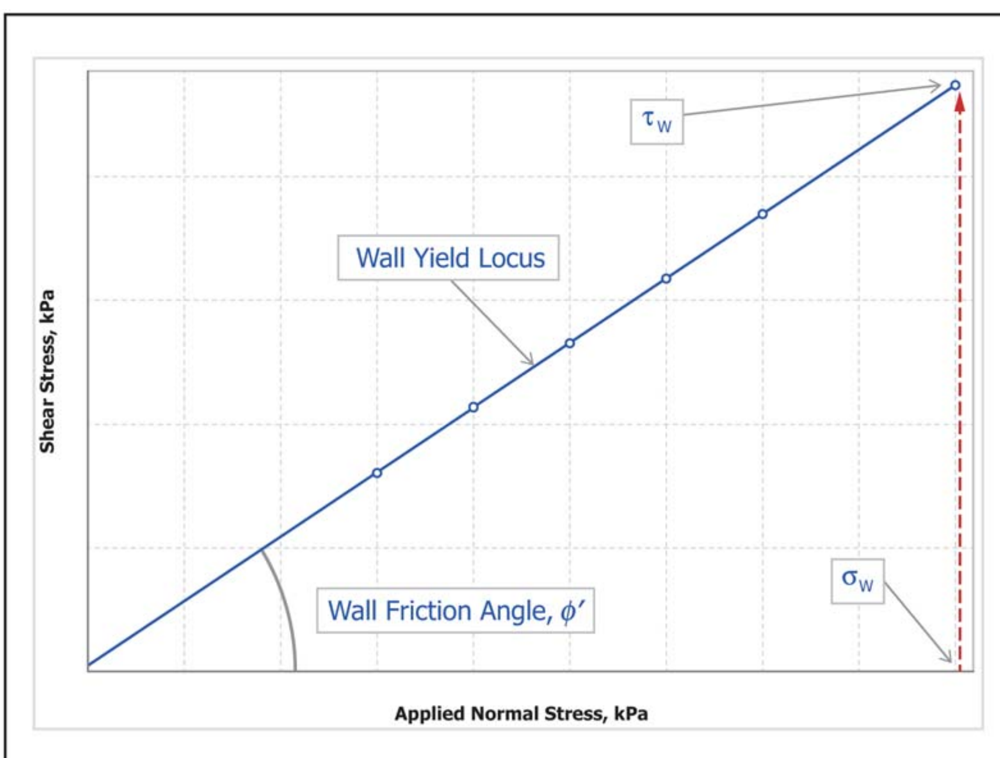

FIG. 11 Graphical Representation of the Wall Yield Locus

This is known as the wall yield locus (WYL), and the angle that is subtends to the x-axis is known as the angle of wall friction, φ'.

Calculate the wall friction angle by:

$$\varphi' = \tan^{-1}\left(\frac{\tau_w}{\sigma_w}\right) \quad (4)$$

where:

φ′ = angle of wall friction (degrees)
σ_w = normal stress at steady state flow (kPa)
τ_w = shear stress at steady state flow (kPa)

10.11.2 In some cases the WYL is not linear and/or may not pass through (or close to) the origin. In these less common instances, then the alternative analysis presented in Test Method D6128 may be employed.

11. Report: Test Data Sheet(s)/Form(s)

11.1 The methodology used to specify how data are recorded on the test data sheet(s)/form(s), as given below, is covered in 1.4.

11.2 Record as a minimum the following general information (data):

11.2.1 Requesting agency or client and/or identifying number for job or project.

11.2.2 Technician name or initials.

11.2.3 Date test was run.

11.3 Record the following test specific information (data):

11.3.1 Generic name of powder tested.

11.3.2 Chemical name of sample, if known.

11.3.3 Specimen moisture (water) content, if determined. Record value to nearest 0.1 %. Indicate method used to determine moisture if not Test Method D2216.

11.3.4 Temperature of specimen to the nearest 1°C.

11.3.5 Humidity of environment where the powder was tested to two significant digits.

11.4 Provide in plot form the following properties as a function of major principal stress, σ_1. Record all stresses to three significant digits and all angles to nearest 1°:

11.4.1 Unconfined yield strength, σ_c

11.4.2 Angle of internal friction, φ.

11.4.3 Effective angle of friction, δ.

11.4.4 Cohesion, C.

11.4.5 Wall friction angle, φ'.

12. Precision and Bias

12.1 *Precision*—Test data on precision is not presented due to the nature of the powder tested by this standard. It is either not feasible or too costly at this time to have ten or more laboratories participate in a round-robin testing program. In addition, it is either not feasible or too costly to produce multiple specimens that have uniform physical properties. Any variation observed in the data is just as likely to be due to specimen variation as to operator or laboratory testing variation.

12.2 Subcommittee D18.24 is seeking any data from the users of this standard that might be used to make a limited statement on precision.

12.3 *Bias*—There is no accepted reference value for this standard; therefore bias cannot be determined.

13. Keywords

13.1 effective angle of friction; effective yield locus; flowability; flow function; Freeman Technology, FT4 Powder Rheometer; internal friction angle; powder; rotational shear cell; unconfined yield strength; wall friction; wall friction angle

ANNEX

(Mandatory Information)

A1. PRE-SHEAR PROTOCOLS AND SHEAR PEAKS

A1.1 Adjusting Pre-Shear Protocols

A1.1.1 There is still some debate as to the appropriate degree of pre-shearing required to achieve critical consolidation prior to undertaking a shear test. Within any FT4 shear test program it is possible to select and adjust the pre-shearing protocols such that the length and number of pre-shears is completely flexible. An automated routine is included as standard to identify a pre-shear peak and move the program onto the next step. This can be disabled and a defined time period/shear distance can be specified. The number of pre-shears can also be specified from a minimum of one, to between two and 100. A criterion to exit a multiple pre-shearing routine based on the convergence of the observed peak shear stresses of the last two pre-shear steps (to a specifiable limit) is also available. Thus it is possible to replicate the methodologies specified in Test Methods D6682 and D6773.

A1.2 Identifying Shear Peaks

A1.2.1 The recommended procedure for the FT4 Powder Rheometer is to undertake multiple pre-shears such that the peak stress of the last two pre-shears are within 1 % of each other or a maximum of ten pre-shear steps have been completed before shear testing is initiated. The shear peaks are automatically identified, and the program steps forward to the next part of the program 20 seconds after a peak has been detected.

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