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Analysis of constant-volume shear tests based on precise measurement of stresses in powder beds

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A R T I C L E   I N F O

A B S T R A C T

This study demonstrates a new constant-volume shear test configuration to analyze the stresses in powder beds and evaluate powder flowability. A novel cylindrical shear cell geometry and load cell arrangement allowed precise measurement of the normal stress acting on the shear planes of the powder beds. The stress transmission ratio between the top and shear planes decreased with increasing ratio of the powder bed height in the upper section of the shear cell to the shear cell diameter. This was due to friction between the powder bed and the side wall of the upper section of the shear cell. Using the measured values of the normal stress on the shear planes, the effects of the powder bed height and shear cell diameter were eliminated from the data. In addition, to evaluate the shear properties of the powder beds, the powder yield locus, consolidation yield locus, critical state line, shear cohesion, and void fraction were obtained from a single shear test. The powder yield locus data were used to obtain flow functions.

1. Introduction

In recent years, particle size reduction has become increasingly popular in various industries to improve the quality and performance of functional particles. However, small particles easily adhere and have low flowability, which causes problems related to powder handling in the development of new products and quality control of industrial processes. Appropriately evaluating powder flowability to resolve these issues remains challenging; many characteristic properties of particles, e.g., particle size distribution, particle density, particle shape, and specific surface area, affect the powder flow behavior in a complicated manner. Consequently, it is difficult to accurately predict the powder flowability even if all relevant characteristics can be quantified.

To quantitatively evaluate powder flowability, various methods and characteristic values have been proposed, e.g., the angle of repose, bulk density, compressibility, tensile strength, and shear strength; however, these values do not always lead to the same results. To comprehensively evaluate powder flowability, Carr [1] proposed a series of indices that correspond to different flow phenomena, i.e., the angle of repose, compressibility, angle of spatula, and cohesion or uniformity. This method is effective for evaluating the powder flowability under low stress. In addition, the avalanche

method [2], vibratory feeder method [3], vibrating tube method [4,5], and vibration shear tube method [6] are effective for similar conditions as the applied forces are rather small.

On the other hand, for large stresses, the flowability depends on the magnitude of the applied stress. Hence, it is necessary to precisely measure such stresses and shear tests have been used for this purpose. Such test methods can be classified into several types depending on the structure of the shear cell, such as the Jenike cell [7,8] and rotational shear cell [9–11]. In addition, standards for the measurement and evaluation methods have been developed [12–15]. These shear tests have been employed in research in various industrial fields, e.g., the food industry, to measure the effect of moisture content [16,17], storage time [18], and particle shape [19,20], on the flowability, and in the pharmaceutical field for tableting [21] and prescription design [22]. The results of such shear tests are often used to design silos and hoppers [23] as a large amount of powder is naturally consolidated by gravity in such applications. In materials research, the flowability of nanoparticles has been analyzed [24].

Several developments have been made to both shear test equipment [25] and analytical methods [26,27], allowing use of the technique in expanded application areas. In the Jenike shear tester, the normal stress on the powder bed is determined by a weight placed on top of the powder and the normal stress on the horizontal cross-sectional area decreases due to the friction between the powder bed and the side wall of the shear cell. Therefore, the normal stress

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Nomenclature

A_P	horizontal cross-sectional area of powder bed (m^2)		γ_{TD}	stress transmission ratio between top and shear planes at steady-state shear, i.e., point D (–)
A_L	area of side of powder bed in lower section of shear cell (m^2)		ε	void fraction (–)
A_U	area of side of powder bed in upper section of shear cell (m^2)		ρ_b	bulk density (kg/m^3)
C	shear cohesion (Pa)		σ	normal stress (Pa)
D_C	inner diameter of shear cell (m)		σ_1	major principal stress given by the Mohr stress circle of steady state flow (Pa)
D_{p50}	mass median diameter of powder (m)		σ_g	geometric standard deviation of particle diameter (–)
F	force (N)		τ	shear stress (Pa)
FF	flow function (Pa)		φ_{CSL}	angle of critical state line (°)
f_c	unconfined yield strength (Pa)			
ff_c	$=\sigma_1/f_c$ (–)		*Subscripts*	
g	acceleration due to gravity (m/s^2)		C	cell
H_{PU}	powder bed height in upper section of shear cell (m)		E	point E (steady-state shear)
k	constant in Eq. (6) (–)		H	horizontal
ΔL_H	horizontal shear displacement (m)		L	lower
M_B	mass of base (kg)		P	powder
M_{BP}	mass of bottom plate (kg)		S	shear plane
M_P	mass of powder (kg)		U	upper
t	time (s)		V	vertical

on the shear plane is not equal to the value calculated simply from the weight and the cross-sectional area. In a previous study [28], we used a constant-volume shear tester and proposed a method for measuring vertical forces acting on both the bottom and top of the shear cell; however, the stresses in the powder beds were not studied in detail.

In the present study, the effect of powder bed height and shear cell diameter on the stresses was investigated and the validity of the constant-volume shear tests based on the normal stress on the shear plane was verified. In addition, the powder yield locus (PYL), consolidation yield locus (CYL), critical state line (CSL), shear cohesion, and void fraction were obtained under various conditions to evaluate the shear properties of the powder beds. Furthermore, the PYL data were used to obtain flow functions.

2. Materials and methods

2.1. Constant-volume shear test apparatus

Fig. 1 shows schematic diagrams of the two types of common shear test methods, i.e., the constant-load and constant-volume methods. The former uses a weight to apply a constant normal stress to the top plane of the powder bed, while the latter uses a mechanical press, where the vertical position of the top plane of the powder bed is fixed during the shear test.

Fig. 2 schematically illustrates the shear stress (τ) obtained from the constant-volume test as a function of the normal stress (σ) and the void fraction (ε) [29]. When shearing at a constant velocity starts from point D, the normal stress decreases and the shear stress increases; however, these stresses approach their respective constant values at point E, which indicates steady-state shear on the critical state line (CSL). After this point, by gradually lowering the base of the shear cell, where there is little change (≤0.5%) in the void fraction of the powder bed, both the shear stress and normal stress decrease (moving along the curve from point E to point C). Therefore, by continuously measuring τ

Fig. 1. Schematic diagram showing two types of shear test methods. (a) Constant-load method. (b) Constant-volume method.
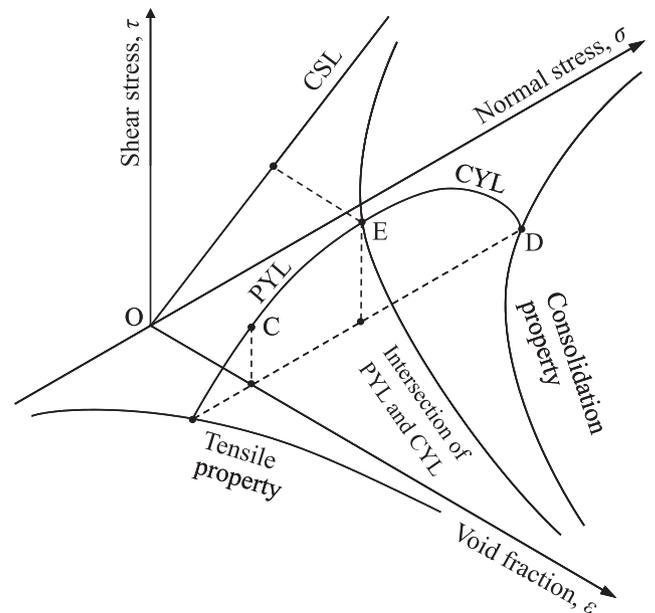
Fig. 2. A three-dimensional diagram showing the relationships between the mechanical properties of a powder bed.

and σ during the shearing process, a relationship between these two factors, i.e., the powder yield locus (PYL), is obtained. The PYL describes the characteristics of the failure of the powder bed under low normal stress. The characteristics of failure under high normal stress are indicated by the curve between points D and E, the consolidation yield locus (CYL). Therefore, complete behavior of the powder bed can be characterized by determining the two yield loci.

2.2. Experimental apparatus

Fig. 3 shows a schematic diagram of the experimental apparatus. The inner diameters (cell diameter D_C) of the stainless steel cylindrical shear cells with separate upper and lower sections were 15, 30, and 43 mm. The cylindrical upper section was fixed and the lower section moved horizontally to induce shearing. The edges of the cylindrical upper section and the lower section were separated by a narrow gap (0.2 mm), and when not filled with powder, the horizontal movement produced a very small amount of friction on the lower section (less than 0.3 kPa, which is thought to be insignificant compared to the stress applied during the shear test). The upper part of the setup featured a vertical servo motor to move the upper layers of the powder bed downward. The vertical force acting on the top of the shear cell, the vertical force acting on the bottom, and the horizontal shear force were measured using load cells. Additionally, the height of the powder bed in the cylindrical upper section and the horizontal shear displacement were measured using non-contact displacement sensors.

2.3. Mechanism for measuring normal and shear stress

Fig. 4 shows a schematic diagram of the cell structure of the shear testing apparatus and the stresses acting upon the powder bed when a vertical load was applied. Fig. 4(a) shows the apparatus with a separation between the bottom plate and the side wall of the lower section of the shear cell, while Fig. 4(b) shows a structure where the lower piece of the cell is used as the bottom plate. In both cases, load cells were located in both the upper and lower sections, enabling the measurement of the vertical force acting on the top of the shear cell (F_{VU}) and the vertical force acting on the bottom of the shear cell (F_{VL}).
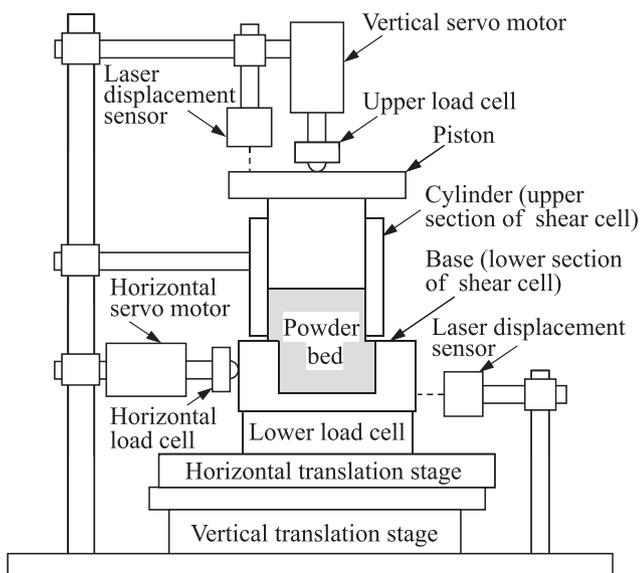
Fig. 3. Schematic diagram showing the experimental apparatus for the constant-volume shear tests.

(a) Separable structure   (b) Non-separable structure
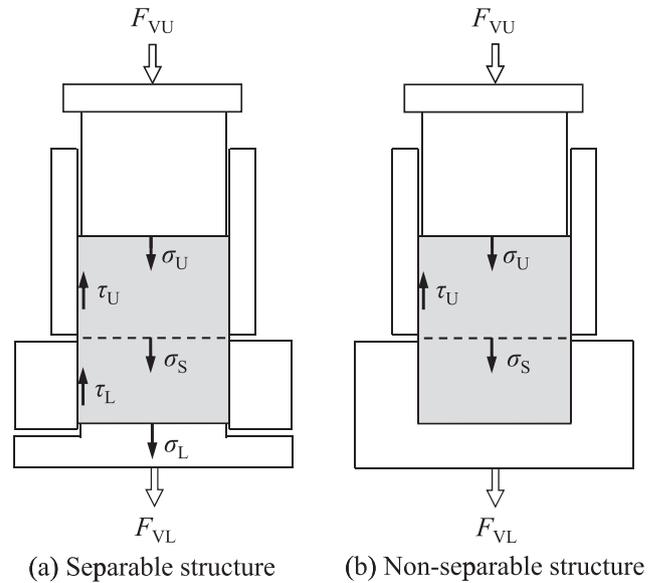
Fig. 4. Schematic diagram showing the two types of shear cells used for constant-volume shear tests.

When the bottom plate is separated, the vertical force (F_{VL}) can be expressed as:

$$F_{VL} = F_{VU} + (M_{PU} + M_{PL} + M_{BP})g - \tau_U A_U - \tau_L A_L, \tag{1}$$

where M_{PU} and M_{PL} are the masses of the powder filling the upper and lower sections, respectively, M_{BP} is the mass of the bottom plate, g is the acceleration due to gravity, τ_U and τ_L are the friction stresses between the powder bed and the side walls of the shear cell in the upper and lower sections, respectively, and A_U and A_L are the areas of the powder bed in contact with the side walls of the shear cell in the upper and lower sections, respectively. Here, the variables τ_U and τ_L in Eq. (1) were not measured.

When the lower section of the shear cell acts as the bottom plate, the vertical force (F_{VL}) is expressed as:

$$F_{VL} = F_{VU} + (M_{PU} + M_{PL} + M_B)g - \tau_U A_U, \tag{2}$$

where M_B is the mass of the base. The variable τ_U in Eq. (2) was not measured, but all other values were known, enabling calculation of this force. The normal stress most relevant to the shear test is not the stress on the top and bottom planes of the powder bed, but rather the stress on the shear plane. The normal stress acting upon the shear plane (σ_S) is expressed as:

$$\sigma_S = \frac{F_{VU} + M_{PU}\,g - \tau_U A_U}{A_P}, \tag{3}$$

where A_P is the horizontal cross-sectional area of the powder bed. By combining Eqs. (2) and (3), the following equation is obtained:

$$\sigma_S = \frac{F_{VL} - (M_{PL} + M_B)g}{A_P}. \tag{4}$$

In other words, when the lower section is used as the bottom plate, the normal stress acting on the shear plane (σ_S) can be calculated from F_{VL}, which means that the relationship between the powder bed shear stress (τ_S) and σ_S can be precisely determined. In this work, we decided to use a cell with the lower section acting as a bottom plate.

2.4. Sample preparation

White fused alumina abrasive (WA, Fujimi Inc.) was used as the powder in the shear test. WA consists of fused alumina pulverized

into fine particles, and is widely used in precision manufacturing. Table 1 shows the mass median particle diameter (D_{p50}) and geometric standard deviation (σ_g) of the WA powder used. The particle shape was irregular.

2.5. Experimental procedure

After loading the sample into the shear cell, the vertical servo motor lowered the top plane of the sample powder bed at 0.2 mm/s to apply pressure to the powder bed. When a set value of σ_S was reached, the movement was stopped and the position was held for a period (maximum 120 s) to relieve the excess stress. During the shear tests, different amounts of powder were filled into the cell to give powder bed heights (H_{PU}) of 10 ± 1 mm, 15 ± 1 mm, and 20 ± 1 mm in the upper section. A horizontal servo motor moved the lower section of the shear cell at a set speed (10 μm/s) to induce shearing of the powder bed. Once the shear stress reached a critical value, the stage upon which the shear cell rested was gradually lowered until σ_S reached zero to complete the shear test.

2.6. Measurements and analytical methods

2.6.1. Time sequence of shear test

Fig. 5 shows representative measurement results obtained using shear tests performed with the apparatus shown in Fig. 4 (b). It shows, from top to bottom, the time dependence of variations in the powder bed height in the cylindrical upper section of the shear cell (H_{PU}), the horizontal shear displacement (ΔL_H), the vertical force measured by the upper load cell (F_{VU}), the vertical force measured by the lower load cell (F_{VL}), and the horizontal shear force (F_H). After the sample was loaded, the process could be divided into four parts: (i) application of stress to the powder bed by the vertical servo motor, (ii) stress relief after stopping the motor and movement, (iii) powder bed shearing induced by the horizontal servo motor, and iv) powder bed shearing induced by gradually lowering the stage upon which the shear cell rests, thus decreasing the vertical force. Points C, D, and E in Fig. 5 correspond to those defined in Fig. 2.

During stressing process (i), the stress was applied to the powder bed until σ_S was reached; friction between the powder bed and the side wall of the shear cell resulted in different magnitudes of F_{VU} and F_{VL}. The stress relieving process (ii) relieved the excess stress in the powder bed by allowing very small movements of the particles; however, after particle movement ceased, F_{VU} was not equal to F_{VL} (point D). In shearing process (iii), the two vertical forces decreased and approached constant values (point E). The horizontal shear force increased up to a certain value due to shearing but then decreased with decreasing vertical force. The purpose of shearing process (iv) was to decrease both the vertical and horizontal forces, where the stage on which the shear cell rested was manually lowered. The variation in the force with time depended on the velocity at which the stage was lowered. However, when the rate of change of normal pressure is less than 10 kPa/s, the shear stress responds well to the normal stress without a time delay. As a result, the velocity of the stage did not affect the

Table 1
Powder properties.

Materials	D_{p50} (μm)	σ_g (−)
Fused alumina #3000	4	1.3
Fused alumina #1200	10	1.2
Fused alumina #280	48	1.2
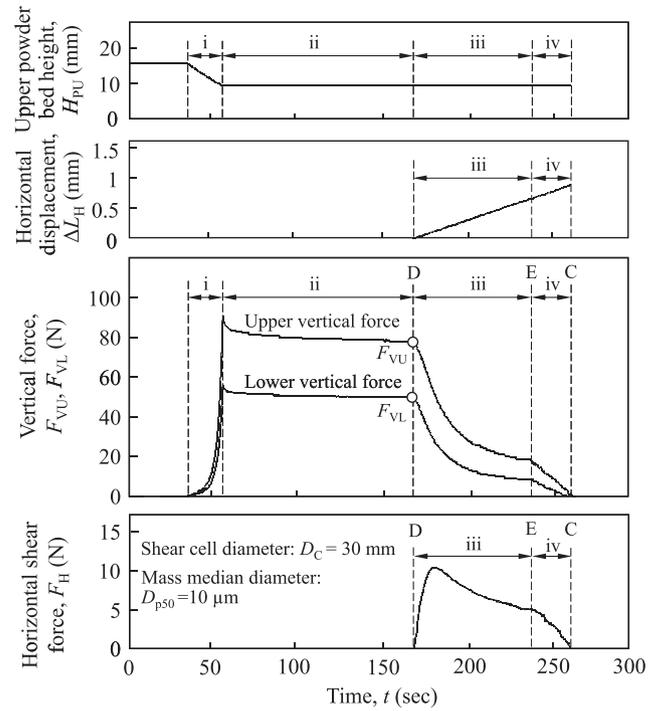
Fig. 5. Time sequence of the shear test.

relationship between the shear and normal stresses, which enabled us to elucidate the inherent properties of the powder.

2.6.2. Stress transmission ratio

In the experimental apparatus used here, the cylindrical upper section of the shear cell remained fixed while the lower section moved. Due to the friction between the powder bed and the side wall of the shear cell, the normal stress on the horizontal cross-sectional area decreased with increasing powder bed depth. Here, the stress transmission ratio between the top plane and the shear plane (γ_{TD}) after stress relief (point D) was calculated from σ_S and the normal stress on the top plane of the powder bed ($\sigma_U = F_{VU}/A_p$), i.e.,

$$\gamma_{TD} = \frac{\sigma_S}{\sigma_U}. \tag{5}$$

Applying Janssen's theoretical model [30] to the powder bed, where the effect of its weight is negligible (less than 1% under the experimental conditions), the stress transmission ratio is expressed as:

$$\gamma_{TD} = \exp\left(-k\frac{H_{PU}}{D_C}\right), \tag{6}$$

where k is a dimensionless constant. Hence, the stress transmission ratio can be calculated from the ratio of H_{PU} to D_C.

2.6.3. CYL, PYL, and CSL

The value τ_S can be determined by dividing F_H by A_p. Further, σ_S can be determined by substituting F_{VL} into Eq. (4). The relationship between τ_S and σ_S over time between points D and E corresponds to the CYL, and the relationship between τ_S and σ_S between points E and C corresponds to the PYL. In addition, the straight line connecting point E and the origin corresponds to CSL.

2.6.4. Flow function

The powder flowability can be evaluated by a flow function (FF) based on the PYL obtained from the shear test [7], which expresses

the relationship between the unconfined yield strength (f_c) and the major principal stress given by the Mohr stress circle of steady state flow (σ_1):

$$ff_c = \frac{\sigma_1}{f_c}, \tag{7}$$

The standards for flowability are the following: $1 < ff_c < 2$: very cohesive; $2 < ff_c < 4$: cohesive; $4 < ff_c < 10$: easy flowing; and $ff_c > 10$: free flowing [26].

3. Results and discussion

3.1. Stress transmission ratio

Fig. 6 shows the relationship between γ_{TD} and H_{PU} for samples with different D_{p50} values. The value of γ_{TD} decreased with increasing H_{PU}. As H_{PU} increased, the area of contact between the powder bed and the shear cell wall also increased; thus, the effect of friction increased, resulting in the decrease in γ_{TD}. This figure also depicts the theoretical curves of Eq. (6), as derived from the Janssen model, showing that the experimental results agreed well with the theoretical calculations. The value of k in Eq. (6) determined by fitting the data was 2 ± 0.3, and depends on the mechanical properties of the powder bed in the shear cell. However, the effect of particle diameter on k is not accounted for in this data.

Fig. 7 shows the relationship between γ_{TD} and D_C for samples with different H_{PU} values. The value of γ_{TD} increased with increasing D_C resulting in a decrease in the effect of friction on γ_{TD}, while the γ_{TD} values decreased with increasing H_{PU}. The experimental data shown in this figure also agreed well with the theoretical curves.

Fig. 8 shows the relationship between γ_{TD} and H_{PU}/D_C. Since these experimental results agreed well with the theoretical calculations based on H_{PU}/D_C, we concluded that the value of γ_{TD} can be evaluated using H_{PU}/D_C. These results show that by decreasing H_{PU} and/or increasing D_C, the normal stress in the upper section of the shear cell is effectively transmitted to the shear plane. It is therefore necessary to consider H_{PU}/D_C when designing the geometry of a cell for experimental shear tests.

3.2. Effect of powder bed height on the YL in the cylindrical section of the shear cell

Fig. 9 shows τ_S as a function of σ_U for different H_{PU} values. To unify the initial condition of shearing, σ_S at the start of shearing was set within a fixed range (215 ± 5 kPa). As a result, τ_S at the

Fig. 7. Effect of the shear cell diameter D_C on the stress transmission ratio γ_{TD}.
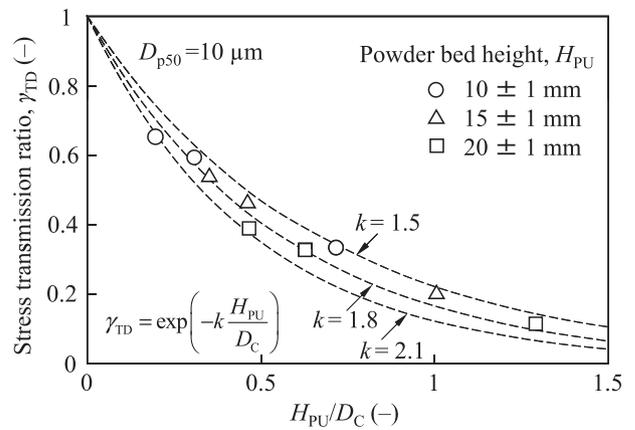
Fig. 8. Effect of the shape of the powder bed H_{PU}/D_c on the stress transmission ratio γ_{TD}.
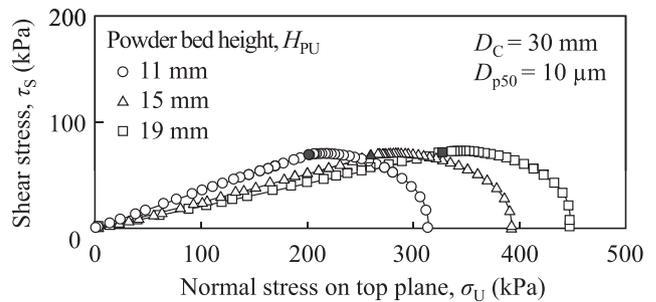
Fig. 9. Shear test results (τ_S–σ_U plots) at different powder bed heights H_{PU}.

Fig. 6. Effect of powder bed height H_{PU} in the upper section of the shear cell on the stress transmission ratio γ_{TD} between the top and shear planes at point D.
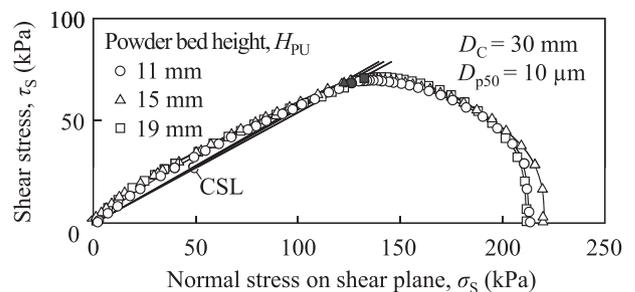
Fig. 10. Shear test results (τ_S–σ_S plots) at different powder bed heights H_{PU}.

critical state indicated by point E (marked by black filled data points) was almost constant (70 kPa) irrespective of H_{pu}. However, for high values of H_{pu}, σ_U must also be high considering friction between the powder bed and the shear cell wall. Therefore, there is a large discrepancy in the observed YLs. Since the normal stress at point E could not be determined, the evaluation of the YL as a function of σ_U was not valid.

Fig. 10 shows the results of plotting τ_S as a function of σ_S for different H_{PU} values; these data were obtained from the same shear tests as those shown in Fig. 9. It was found that the YL and CSL values were similar for all H_{PU} values. Therefore, even when shear tests were conducted with different powder bed heights, analysis of τ_S as a function of σ_S is an appropriate method for accurate evaluation of the powder properties.

3.3. Effect of the cell diameter on YL

Fig. 11 shows τ_S as a function of σ_U for different D_C values. The initial σ_S value was set within a fixed range (110 ± 3 kPa). Since the area of contact between the powder bed and the shear cell wall varied according to the magnitude of D_C, the effect of friction and the transmissibility of the normal stress also depended on D_C. In other words, when the cell diameter decreased, the stress transmission ratio also decreased due to the increase in the effect of friction between the powder bed and the shear cell wall. This means that for low D_C values, a higher σ_U value is required to achieve the same σ_S value, resulting in a large discrepancy in the observed YL values.

Fig. 12 shows τ_S as a function of σ_S for different D_C values, which were obtained from the same shear tests as the data shown in Fig. 11. These YL and CSL values were similar for all cell diameters. Therefore, proper evaluation of the powder properties is possible by conducting shear tests and analyzing τ_S as a function of σ_S, irrespective of the D_C value.

3.4. Effect of the particle diameter on YL

Fig. 13 shows the relationship between τ_S and σ_S for two types of powder with different D_{p50} values (10 μm and 48 μm). We set σ_S to three different levels and obtained the corresponding PYL and CYL values. The void fraction of the powder beds (ε) depended on the initial normal stress, so it needed to be measured for each shear test. YL varied depending on ε; the value at the steady-state shear (point E, marked by filled data points) also varied. However, all E points fell along the CSL intersecting the origin. The angle of the CSL corresponds to the angle of dynamic friction, i.e., $\varphi_{CSL} = 31°$ for $D_{p50} = 10$ μm and $\varphi_{CSL} = 25°$ for $D_{p50} = 48$ μm. This demonstrates that smaller particles show a higher angle of dynamic friction, resulting in poorer powder flow.

3.5. Flow function

Fig. 14 shows the flow functions (solid lines) derived from the results in Section 3.4 and lines of constant flowability ($ff_c = 1, 2, 4,$ and 10, broken lines). The flow function curve for smaller particles exhibited smaller ff_c values, i.e., lower flowability. Here, it should be noted that using the present constant-volume shear test configuration, we could obtain YL from a single shear test; thus, the flow function can be derived much more easily than demonstrated previously. For reference, the relevant data, i.e., the bulk density ρ_b, void fraction ε, shear cohesion C, angle of dynamic friction of powder φ_{CSL}, major principal stress σ_1, unconfined yield strength f_c, and the ratio ff_c (=σ_1/f_c) derived from the shear tests for the sample powder, are shown in Table 2.
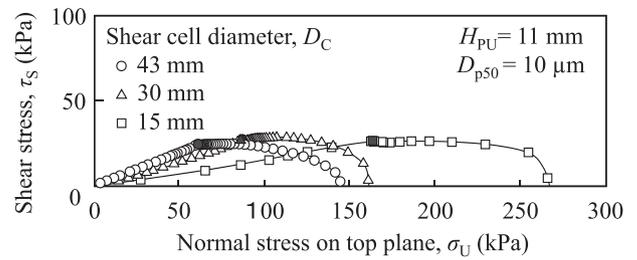
Fig. 11. Shear test results (τ_S–σ_U plots) at different shear cell diameters D_C.
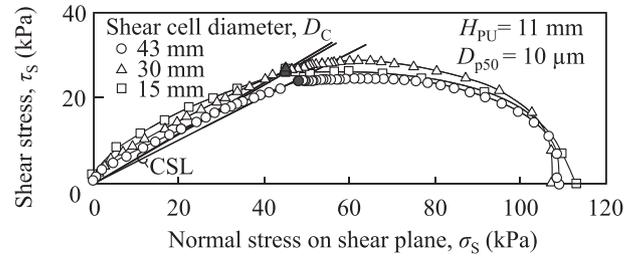
Fig. 12. Shear test results (τ_S–σ_S plots) at different shear cell diameters D_C.
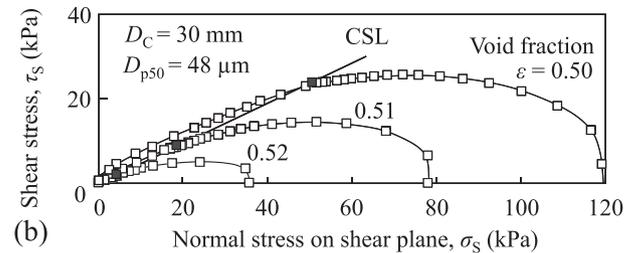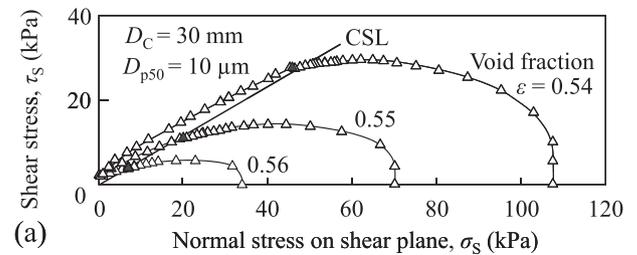
Fig. 13. Shear test results (τ_S–σ_S plots) at different void fractions ε. (a) $D_{p50} = 10$ μm and (b) $D_{p50} = 48$ μm.
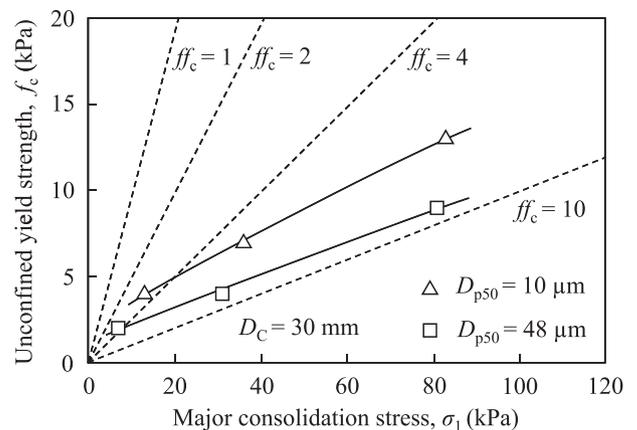
Fig. 14. Flow functions (f_c–σ_1 plots) of powders and lines of constant flowability ff_c.

Table 2
Summary of experimental results for shear tests of powders.

	ρ_b (kg/m³)	ε (–)	C (kPa)	φ_{CSL} (°)	σ_1 (kPa)	f_c (kPa)	ff_c (–)
Fused alumina (#1200)	1700	0.56	2.3	31	13	4	3.3
D_{p50} = 10 μm	1750	0.55	1.8		36	7	5.1
	1790	0.54	2.0		83	13	6.4
Fused alumina (#280)	1860	0.52	0.8	25	7	2	3.5
D_{p50} = 48 μm	1910	0.51	0.8		31	4	7.8
	1950	0.50	0.7		81	9	9.0

4. Conclusions

Here we demonstrated a constant-volume shear test apparatus that was able to precisely measure the upper and lower normal stresses of powder beds that allowed us to investigate powder flowability. Using the lower section of the shear cell as the bottom plate enabled measurement of the normal stress on the actual shear plane rather than the normal stress on the top or bottom plane of the powder bed. As the ratio of the powder bed height in the upper section of the shear cell to the shear cell diameter increased, the stress transmission ratio decreased. Evaluating the normal stress on the shear plane, the effects of the shape and/or size of the shear cell were eliminated from the results and reproducible powder flowability behavior was observed.

The proposed experimental method allowed the powder yield locus, consolidation yield locus, critical state line, shear cohesion, powder bed void fraction, and stress transmission ratio to be easily and precisely measured with a single shear test. In addition, shear test data at three levels of normal stress were efficiently obtained, making this method suitable for evaluating the flow function.

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