Discontinuous Flow and Frictional Property of Granular Materials

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Abstract

The stress-strain relation in the deformation and flow of granular materials is generally discontinuous. The discontinuous behaviour is one of the fundamental properties of granular materials. The mechanism of discontinuous behaviour in the flow of granular materials is investigated by the penetration of a circular plate into a powder bed. The penetration characteristic curve (load-displacement curve) of a dense powder bed shows a step-wise curve. The load required to penetrate a circular plate into the powder bed is in satisfactory agreement with the values calculated from Meyerhof's equation. The plasticity pattern formed by the penetration of a plunger into the powder layer is observed by an X-ray radiograph. It is shown that discontinuous behaviour results in successive or periodical shear yields which occurred in the powder bed. The generating mechanism of intermittent or periodical shear yields is theoretically discussed, and the relationship between the periodicity of intermittent shear yields and the frictional property of the powder bed is experimentally confirmed. The static and dynamic angles of the internal friction of powders can be measured by the penetration test.

1. Introduction

The stress-strain relation in the deformation and flow of the powder layer constructed by the particles contacting each other generally indicates discontinuous curve1). The discontinuous behaviour is one of the important fundamental properties of the powder layer in which the force is transmitted by the friction through the contact points of the particle. There are thus various phenomena caused by the discontinuity, such as the pulsation of the flow rate of the granular materials from the hopper and the wall pressure, the heterogeneity of the density within the compressed granular materials and the periodical fluctuation of the power required for powder mixing. Hence, the fundamental study on the discontinuous behaviour in the deformation and flow is of great importance for the powder handling.

For the purpose of elucidating the discontinuous flow property, the authors paid attention to the compressive flow caused by the penetration of a circular plate into a powder layer, of which the dynamic analysis could be conducted rather simply. Based on the experimental study on the relation between the penetration load and the displacement of the plate, the discontinuous behaviour has been investigated in relation to the frictional properties1).

In this paper, the propagating condition of

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the plasticity in the penetrating process within a relatively dense powder layer was observed by means of X-ray radiograph minutely. It verified that the load-displacement curve in the penetrating process showed a step-wise variation by the flow of granular materials which consisted of successive or periodical shear yields. Furthermore, the mechanism of generating the discontinuous behaviours was theoretically discussed on the basis of the observation of the forming process of the plasticity pattern. The measurement of the frictional angle property by the penetration method and its physical meaning will be also reported in the following.

2. Experimental apparatus and method

2.1 Measurement of penetrating load and displacement

Figure 1 shows a schematic diagram of the measurement apparatus. The loading test apparatus (Model UMH-10 made by Shimadzu-seisakusho) was used to load the penetrating circular plate at a constant rate. The sample powder was fed through a sieve and uniformly packed in the cylindrical sample container, which was then set on the movable stage of the loading test apparatus. Raising the movable stage at a constant rate, the penetrating circular plate contacted with the surface of the powder layer and started to penetrate. The resistance force (load) acting on the penetrating plate from the powder layer and the depth of the penetrating plate (displacement) from the surface of the powder layer were measured by the load cell and the displacement transducer respectively. The cylindrical sample container (φ 298 x H111) was made of PVC resin. It was necessary to make the frictional force on the axis supporting the penetrating plate and its periphery negligibly small compared with the load acting on the bottom of the penetrating plate from the powder layer. The penetrating circular plates having a diameter 30², 20 and 14.4 mm with the thickness of 5 mm were used and supported by the metal cylinder of a diameter of 4 mm. The measurements were conducted at constant penetrating velocities as well as at constant loading rate by setting a spring having a constant of 1.16 N/mm above the penetrating plate.

2.2 X-ray observation of plasticity pattern within powder layer

In order to estimate the penetrating load into the powder layer and understand the deformation and flow mechanism, it is necessary to observe minutely the momentary plasticity pattern formed in the powder layer during the penetration of the plate. Figure 2 shows a schematic diagram of the apparatus used for the observation. This consisted of the transparent box made of acrylic resin (430 x 470 x 50 in mm), the plunger (25 x 48 x 500 in mm) having the rectangular cross section, the motor for its penetrating into the powder layer at a constant speed and X-ray radiograph equipment. The way of filling the box with the sample was the same as mentioned above. Additionally, steel balls with a diameter of 3 mm were arranged in the lattice form having a side of 10 mm in the central plane vertically oriented to the X-ray radiation, to measure the displacement of the particles on the deformation and flow. Having the plunger penetrate at a constant velocity, the load and the displace-
ment were measured by the above-described method. At the same time, the X-ray radiographs of the plasticity pattern inside the powder layer were obtained using the intermittent pulses having a duration of 240 seconds from an X-ray source (ERESCO-300 operated at a tube voltage of 140 kV and a tube current of 5 mA) and the X-ray film (Fuji Film IX-100 for industrial use, 300 x 250) attached to a side wall of the powder layer opposite to the one irradiated by the X-rays. Furthermore, the instantaneous change in the plasticity pattern was recorded and observed continuously with an X-ray video system (Rikagaku Denki).

2. 3 Sample

In this paper a silica sand was used as a sample to distinguish the discontinuous behaviour on the deformation and flow. The silica sand used for the X-ray observation of the plasticity pattern had an average particle size of 350 μm and the angle of internal friction of 35° measured by a direct shear test. On the other hand, an Ottawa sand classified into the range from 297 to 500 μm with JIS standard sieve (silica sand A) was used for the penetration test. In order to obtain a sample which was made of the same material as the silica sand A but differed in the internal friction, a silica sand B was prepared by washing sufficiently the particle surface of the silica sand A with pure water. The angles of internal friction $\phi_a$ of the silica sands A and B by the direct shear test were $\phi_a = 39°$ and $\phi_b = 41°$ respectively, both of them having $\rho_b = 1.700 \text{ g/cm}^3$. White corundum (WA #3000, $D_p = 5 \mu m$) was also used as a fine powder sample.

3. Results and discussions

3. 1 Penetration characteristic curve

The solid line in Fig. 3 shows an example of the penetration characteristic curve which represents the relationship between the load and...
the displacement obtained from the penetration tests at a constant speed. It is apparent from Fig. 3 that the penetration characteristic curve is affected by the penetrating speed. Therefore decreasing the penetrating speed as far as less than 0.1 mm/s, the following experiments were conducted under the condition regarded as the quasi-static state. Consequently, the load required for the plate to penetrate the powder layer \( Q_a \) equaled so-called ultimate bearing capacity on the analogy of the bearing capacity problem in the soil mechanics.

It has been much debated about the ultimate bearing capacity of the circular foundation. Having applied here the semi-theoretical bearing capacity equations of Terzaghi and Meyerhof as expressed in the following equations, the calculated results for the penetrating plate (the circular foundation) are shown by broken lines and chain lines with the frictional angle \( \phi \) as a parameter in Fig. 3\(^2,3\).

Terzaghi's equation

\[
Q_a = 1.3 C N_e + \rho_B h N_q + 0.6 \rho_B (B/2) N_r
\]

(1)

Meyerhof's equation

\[
Q_a = 1.3 C N_e d_c + \rho_B h N_q d_q + 0.6 \rho_B (B/2) N_r d_r
\]

(2)

where \( C \) is the cohesive force, \( \rho \) is the bulk density, \( B \) is the diameter of the penetrating plate, \( h \) is the displacement and \( N_e, N_q, N_r \) are the coefficients of the bearing capacity given as a function of the angle of internal friction.

\[
N_q = \frac{1}{(1 - \sin \phi)} \exp \left\{ \frac{3\pi}{2} - \phi \right\} \tan \phi
\]

\[
N_e = (N_q - 1) \cot \phi
\]

\[
N_r = (N_q - 1) \tan 1.4 \phi
\]

(3)

The coefficients \( d_c, d_q, \) and \( d_r \) considering the effect of the penetrated depth are represented by the following equations.

\[
d_c = 1 + 0.4 (h/B), \quad d_q = 1 + 0.2 (h/B)
\]

\[
d_r = 1 + 0.6 (h/B)
\]

(4)

Consequently if the compacting condition (the bulk density \( \rho_B \)) of the sample is decided, Eqs. (1) and (2) become the function of \( \phi \) and \( h \) and are solved to give the broken and the chain lines in Fig. 3. On this experimental condition, the results from Eqs. (1) and (2) differ considerably as the displacement exceeds 5 mm.

As seen in Fig. 3, the measured value\(^*\) of the load changes according to the calculated value by Eq. (2), as far as the displacement reaches a certain value represented by the point a. Beyond the point, however, the measured load comes off from the curve obtained by the Meyerhof's equation considerably and suddenly increases\(^*\). This is caused by the fact that Eq. (2) was developed for the shallow foundation \( (h < B) \) and that the actual load includes the passive resistance due to the sample container wall. Actually solving Kotter's equation for the axially symmetrical system\(^4\) with the experimental condition given in Fig. 3 produces the slip line at the point a, the top of which reaches the wall of the container. However the trend of the change of the penetration curve up to the point a which is not affected by the wall is consistent with that of the bearing capacity obtained by Meyerhof's equation. Furthermore the shape of the penetration curve is not continuous but step-wise or serrated with alternated increase and decrease of the load as reported previously\(^1\). This feature becomes clearer if the constant load rate test is done as shown in Fig. 4. It is worthy of noticing that each curve enveloping the upper and the lower ends of the steps of the penetration curve obeys the tendency of Meyerhof's equation as shown by the broken line in Fig. 4.

3. 2 Plasticity pattern formed within the powder layer on the deformative flow process

The deformative flow process within powder layer has been analyzed previously by Bagnold's model\(^1\), which included some problematical points. Therefore the momentary change of the plasticity pattern formed within a powder layer in the flow process was observed in detail by the visualization using the X-ray and the

\(\text{\* The load on the abscissa is given as a value per unit area of the penetrating plate i.e. the normal stress on the powder layer under the plate (Pa).}\)

\(\text{\*\* The penetrating load on the surface of the powder layer represented with the solid line in Fig. 4 also does not agree with the bearing capacity obtained by Meyerhof's equation. This is attributed to various reasons, one of which seems to be the compaction of the powder layer near the surface caused by flattening it with a scraper after filling the container with the sample. Therefore, the data in the neighbourhood of the surface were neglected.}\)
flow mechanism of the powder layer was attempted to make clear.

Figure 5 shows a typical example of the X-ray radiograph indicating the state of the plasticity within the powder layer generated by the penetration of the plunger. On the radiographs, the slip lines formed from the top of the plunger toward the circumference are remarked as the white strips. It can be confirmed that these white strips are the slip lines from the measurement of the displacement vectors of the steel balls (the black circles in the pictures) set to observe the displacement of the particles within the powder layer.

Figure 6 shows the displacement vector of the steel balls with arrows, after the plunger penetrated 60 mm from the surface of the powder layer (corresponding to Fig. 5(c)). The numerical figure represents the ratio of the initial area of the grid section formed by four adjacent steel balls to the area enveloped by the same balls after the penetration. Consequently the figure expresses the degree of local expansion or contraction of the powder layer. The broken lines in Fig. 6 show the white strips in Fig. 5(c). As the figures in the area enveloped by the broken lines represent the value more than 1.0, it is confirmed that they are the slip lines with the local expansion. Since this slip line has rather wide width, it is to call the slip belt. The specifically large dilatation in these parts is caused by the following factors. Noting the lowest slip lines formed just at the time when the X-ray radiograph was taken, quantity of the particle displacement over the slip line becomes maximum near the slip line and gradually decreases toward the surface of the powder layer. Moreover, paying attention to the slip lines on the left side of the plunger, the displacement vectors of the particles over the slip line have the clock-wise rotational component to the slip line, while those under the slip line have the counterclock-wise rotational component, even if they are small. As a result, the local large dilatation takes place at the part of
Fig. 6 Measured displacement of steel ball showing the evolution of dilatancy localization

Fig. 7 X-ray radiographs of rupture layer in powder bed
The slip line, which leads to the increase of the X-ray transmission and appears as the apparent white strip on the radiograph reversely developed.

A series of X-ray radiographs in Fig. 5 distinctly shows how the slip lines appearing in the white strips were formed intermittently or periodically as the plunger penetrated.

Figure 7 shows the X-ray radiographs before and after a step-wise change in the penetration process curve formed by the increase and the following radical decrease of the load. That is to say, Figs. 7(a) and (b) indicate the X-ray radiographs as the load was close to the ultimate bearing capacity and as the load suddenly decreased respectively. A part of the slip line (a part of the transitional area) has been formed already right under the plunger in Fig. 7(a) and the top of the slip line reaches the surface of the powder layer with the large particle displacement in Fig. 7(b). From this result, it is understood that each step of the penetration characteristic curve corresponds to each slip line in the powder layer.

3.3 Shape of slip line

Terzaghi's and Meyerhof's equations are obtained by the environing transition zone acted on the rigid zone under the plunger and the passive resistance from Rankine's passive zone as the plasticity on the ultimate state is assumed. The slip lines of the surface in the powder layer obtained by the numerical calculation of Meyerhof's and Kötter's equations are compared with that observed by X-ray in Fig. 8. Three slip lines appear to be consistent as a whole expect the difference of these top patterns.

As the load was added on the powder layer by the plunger, a transition zone was observed to take place in part under the condition of the ultimate bearing capacity. It is possibly considered that a slight difference of the top pattern of each slip line would hardly effect the bearing capacity and the tendency of the load against the displacement could be consistent with that of Meyerhof's hypothesis.

3.4 Generation mechanism in discontinuous behaviour of powder layer

From the fact as mentioned above, the generation mechanism of the discontinuous flow within a powder layer can be described by the following Meyerhof's hypothesis. Consider, for simplification, that the penetration plate penetrates into the infinitely wide and dense powder layer at the fixed displacement rate as shown in Fig. 9. The resistance force, or the load $Q_u(\phi_s, h)$, depending on a static angle of internal friction and a displacement $h$ acts the penetrating place, when it is placed at a given depth $h$ within the powder layer. As the powder layer exhibits the ultimate bearing capacity, a part of the slip line represented by the solid line in Fig. 9 is formed. Then, the slip line reaches the surface of the powder layer accompanied by the movement of the particles toward over the slip line. At this time, the dilatation of the particles over the slip line causes decrease of the static angle of internal friction $\phi_s$ and it becomes the dynamic angle of internal friction $\phi_d$. Thus, the resistance force $Q_u(\phi_s, h)$ of the powder layer decreases $Q_u(\phi_d, h)$ at once. As $\phi_d$ decreases gradually with a dilatation of the powder layer, the penetrating plate penetrates by the displacement $\Delta h$ until the
active zone (rigid zone) is formed. At this time, the bottom angle under the penetrating plate is \(\pi/4 + \phi_e/2\) and the penetrating plate meets resistance of gradually decreasing \(Q_u(\phi_d, h)\). The value, \(\phi_e\) is related to the penetrating rate because \(\phi_e\) is the dynamic angle of internal friction of the powder layer flowing over the slip surface at a constant penetrating rate \(V_p\). The displacement \(\Delta h\) is given by \(\phi_s\) and \(\phi_e\) through the following equation.

\[
\Delta h = \frac{B}{2} \tan \left( \frac{\pi}{4} + \frac{\phi_s}{2} \right) - \tan \left( \frac{\pi}{4} + \frac{\phi_e}{2} \right)
\] (5)

Where \(B\) is the diameter of the penetrating plate.

As the bottom angle of the active zone becomes \(\pi/4 + \phi_e/2\), the dilatancy motion of powder over the slip line stops and the ultimate bearing capacity \(Q_u(\phi_s, h + \Delta h)\) at the depth \(h + \Delta h\) of the powder layer acts on the penetrating plate. Then the new slip line \(A'B'\) is formed as shown by the chain line in Fig. 9. The penetrating plate penetrates into the powder layer with repeated this intermittent behaviour.

As the constantly increasing load through the spring is acted on the penetrating plate, the penetration at a constant rate can be observed as shown in Fig. 10. When the increasing load is equal to the ultimate bearing capacity at any depth \(h\) within the powder layer, the powder layer flows and the penetrating plate suddenly penetrates into the powder layer suffering from the decreasing resistant force \(Q_u(\phi_d, h)\). The penetrating plate stops at the place where the equilibrium of the following equation is achieved.

\[
Q_u(\phi_s, h) - f = Q_u(\phi_e, h + \Delta h)
\] (6)

where \(f\) is the decrease element of the load which is caused by the extension of the spring and \(\Delta h\) is given by the following equation.

\[
\Delta h = \frac{1}{k} Q_u(\phi_e, h + \Delta h) - Q_u(\phi_s, h)
\] (7)

where \(k\) is a spring constant. Then, the penetrating plate is at the rest until the load of \(Q_u(\phi_s, h + \Delta h)\) is acted, and the next flow was repeated.
Figure 11 illustrates the penetrating behaviour with the intermittent flow expressed as the penetration characteristic curve. The penetrating plate penetrates, suffering from the resistant force of \( Q_u (\phi_s, h) \) and \( Q_u (\phi_e, h) \) from the powder layer. When \( \phi_s \) differed largely from \( \phi_e \), the powder layer under the penetrating plate dilates considerably. At this time, the structure of the powder layer is fluctuated and the passive zone based on the smaller angle of internal friction than \( \phi_s \) is formed. Then the slip line (b) which the top of slip line intersects with the slip line (a) is formed as shown in Fig. 6. As a matter of course, the load is small at this time.

3. 5 Measurement of property of frictional angle by penetrating experiment

Discussion of the flow mechanism in the latest section implies that the static and dynamic angle of internal friction within the powder layer can be measured by the penetrating experiment. The top and the under load on each step of the penetration characteristic curve are expressed by \( Q_u (\phi_s, h) \) and \( Q_u (\phi_e, h) \) respectively, in Fig. 11. The values \( \phi_e \) and \( \phi_s \) can be obtained by the penetration characteristic curve enveloped at the top and the bottom of steps respectively, through the penetration characteristic curve corresponding to the angle of internal friction obtained by Eq. (2) and the measured penetration characteristic curve.

The measured result is shown in Fig. 12. For silica sand (a) \( \phi_s \) and \( \phi_e \) obtained under the constant displacement rate condition were 40.7° and 40.4° respectively and under the constant loading rate condition were 41.7° and 40.2°. For silica sand (b) \( \phi_s = 42.3° \) and \( \phi_e = 39.8° \) were obtained at the constant loading rate. Each \( \phi_s \) was similar to the result of the direct shear test approximately. As previously reported\(^1\), \( \phi_s \) and \( \phi_e \) of cohesionless powder were consistent with the angle of repose measured by the method of the inclined container of the powder with the same porosity.

For any powder \( \phi_s \) obtained under the constant displacement rate condition was apt to become lower than that under the constant loading rate condition and conversely \( \phi_e \) became higher. This is probably due to large deviation from the ideal whole shearing condition, as \( \phi_e \) became much smaller compared with the case of the constant loading rate test as mentioned below. The value \( \phi_e \), depending on the penetrating rate, took small value under the constant loading rate condition, because the penetrating rate of the flow had large value. Under the constant loading rate condition, \( \Delta h \) of the silica sand (a) and (b) were 2.2 and 3.1 mm respectively through the calculation of Eq. (7). These values were consistent with the measured result (average \( \Delta h \) were 1.96 and 3.20 mm, respectively). Figure 13 shows the case of the smaller diameter of the penetrating plate (area ratio was 0.52). The result of the penetration test was very sensitive to the porosity \( e \) (bulk density \( \rho \)) of the powder layer.
and the value of $\phi_s$ and $\phi_e$ were a little different from the case of Fig. 12, and $\Delta h$ took 0.84 mm which was about half of the silica sand (a) shown in Fig. 12 as predicted from Eq. (7). Figure 14 shows the influence of the spring constant. The calculated values corresponding to the spring constant 1.022 and 0.706 N/mm were 0.87 and 0.99 mm, respectively, and the average values of $\Delta h$ of the measured result were 0.76 and 0.94 mm, respectively. Therefore Eq. (7) was found to be almost satisfied.

On the other hand, $\Delta h$ obtained at the constant displacement rate was calculated from Fig. 12 and Eq. (5) to be 0.20 mm. This value gives a question for the rupture model as mentioned above because $\Delta h$ took a small value corresponding to a particle diameter of the sample powder. Figure 15 shows the result of the constant displacement rate test of the silica (b) having the same particle diameter. Then $\phi_s$ and $\phi_e$ is 45.8° and 44.4° respectively and 0.61 mm was calculated from Eq. (5). This means that $\Delta h$ would depend on $\phi_s$ and $\phi_e$, independently of the particle diameter. Figure 16 shows a part of the waveform which is indicative of the fluctuation of the load for the test in Fig. 15. Similar to the constant loading rate test, this test provided a periodical rapid decrease showing the failure at intervals nearly equal to the calculation (0.61 mm) as mentioned above. This implies that the model as described above would be available.

When the load is applied under the displacement rate condition, it abruptly decreases, even to a non-loading state, at the powder layer deformation as shown in Fig. 16. As the load increases up to the point a, the particles near the slip line begins to flow and the succeeding collapse takes place. In this work, the load was determined from the intersecting point b of the tangential line (dotted line) and the load curve at collapse of the powder layer to obtain the penetrating load curve related to $\phi_s$. 

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Fig. 13 Penetration characteristic curve of silica sand (a)

Fig. 15 Penetration characteristic curve of silica sand (b) for displacement control test

Fig. 14 Influence of the spring constant on penetration characteristic curve

Fig. 16 Representative waveform of the load required to penetrate a circular plate into powder bed
The above results indicate the measurement example on the property of the angle of internal friction and confirm suitability of the flow mechanism as mentioned in the former section. Although nothing is referred to in detail in this report, a fine powder was found to be packed loosely into a container due to its cohesiveness and to tend to cause, so called, propagating rupture at a surface of the powder layer as shown in Fig. 17. Therefore this test should be done after the dense packing was achieved in such a condition that the large deformation period disappears. The same slope of the penetration load curve calculated from Eq. (2) with the variation of the cohesive force and the measured penetration characteristic curve can easily provides $C$ and $\phi$.

4. Conclusion

When a powder layer deforms or flows under the influence of an external force, its displacement varies discontinuously in general with the added stress. This discontinuous change causes various kinds of trouble in actual operations.

This report dealt with phenomena of plunger penetration into dense powder layer to elucidate the generation mechanism of such a discontinuous behaviour. The results obtained are as follows:

- Penetrating load varied with penetrating displacement in a discontinuous step-wise manner. This means that the powder layer would deform discontinuously. The relation between penetrating displacement and penetrating load obtained by experiment was consistent with the Meyerhof hypothesis.

- X-ray radiograph observation was carried out to obtain the plasticity patterns in the powder layer formed by penetration of the plunger. The result was that a set of slip lines could be formed in an intermittent, periodical manner; thus the penetration process was expressed as a discontinuous curves.

- The Meyerhof plasticity model was adopted to quantitatively elucidate how the extent of its discontinuity would be dependent upon powder characteristics, especially friction property.

- Further, based on such an experimental result, characteristics of friction angle were determined by use of the penetration test.

The authors intend to explore the dependence of penetration characteristic curve on penetrating rate and the relationship between the degree of discontinuity in the curve ($\Delta \phi$ or the difference of $\phi_s$ and $\phi_r$) and the various powder behaviour.

**Nomenclature**

- $B$: diameter of circular penetrating plate [mm]
- $C$: cohesive force of powder [Pa]
- $D_e$: diameter of sample container [mm]
- $D_p$: particle diameter [$\mu$m]
- $N_c, N_q, N_r$: coefficient for estimation of the load required to penetrate a circular plate into powder bed [–]
- $Q_u$: load required to penetrate a circular plate into powder bed (bearing capacity of powder bed) [N]
- $V_p$: penetrating speed of a circular penetrating plate [cm/s]
- $d_c, d_q, d_r$: coefficient on displacement of penetration [–]
- $k$: spring constant [N/mm]
- $h$: displacement of penetration plate from free surface of powder bed [mm]
- $\phi$: angle of internal friction [deg]
- $\phi_i$: angle of internal friction measured by direct shear test [deg]
- $\phi_d$: transient dynamic angle of internal friction of powder bed in collapsing [deg]
- $\phi_e$: dynamic angle of internal friction [deg]
- $\phi_s$: static angle of internal friction [deg]
- $\rho_B$: bulk density [g/cm$^3$]

**References**

1) Hidaka, J. and S. Miwa: Kagaku-Kougaku Ronbunshu, 7 [2] 184-190 (1981).
Explanation of the cover photograph

Hosokawa Micron Corp. has developed recently a fine-grinding equipment, called ANGMILL, which involves new grinding mechanisms. This equipment is designed to be available for mechanochemical applications such as homogenization (precise mixing), surface improvement (coating), and shape transformation, as well as fine grinding.

The left side of the cover photograph shows ground polymer particles which have very complicated shapes. The right side of it, on the other hand, indicates the same particles after treatment by ANGMILL; they look very spherical (about 20 μm in average particle diameter).