Yield Parameters for Iron Powder Compaction on Different Particle Size

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Abstract
Shear failure yield line in Drucker-Prager yield surface for iron powder on different particle size and amount of interparticle lubricant has been estimated. Critical stress state and uniaxial failure stress state have been determined by a single shear test and uniaxial failure test for a green compact. Compaction cap parameter has been estimated by the lateral force measurement test by instrumented die compaction system. By those estimations, materials input parameters for the Drucker-Prager Cap (DPC) model have been determined successfully, and differences in particle size and amount of lubricant can be recognized. Finite element method (FEM) analysis of simple closed-die compaction has been conducted. As a result, the materials input parameters for the DPC model on different powder properties have been successfully identified as the FEM analysis fairly predicts compaction pressure and density distribution on uniaxial compaction.

1. Introduction
Metal powder die compaction is widely applied as a forming method for several sintered parts such as gear, synchronizer hub and so on. It is necessary to control the compression tool velocity that accomplishes uniform consolidation until objective shape dimension of the green compact, and to avoid crack occurrences such as slip-crack [1, 2] and shear failure, but it is not easy to optimize especially in case of highly complex shape with high density. Therefore, it is effective to apply the numerical simulation for powder die compaction process to predict density distributions and shear failure occurrence [3-8]. The Constitutive equation of metal powder is complex because both consolidation yield properties and shear failure yield properties must be considered. The compaction yield function is expressed by elliptical function such as Shima-Oyane model [9]. On the other hand, the Drucker-Prager model [10] which is expressed by linear function is the simplest shear yield function for granular materials. Drucker-Prager Cap (DPC) model is defined as a combination of these functions which is indicated in the principal stress space as conical shape that the tip is closed with a consolidation cap, and its central axis is hydrostatic pressure axis. It is necessary to conduct a triaxial compaction test to estimate the cap parameters in the DPC model [11-14]. However, the testing device is not easy to construct due to the complicated tool moving mechanism. Also, more than two kinds of shear failure
test for green compact must be performed to estimate shear yield parameters in the model. Thus, it takes considerable effort to identify the materials parameters for the \textit{DPC} model. So, we have suggested applying single shear test which is general strength-test in soil mechanics to the iron powder green compact [15] and succeeded in measuring Critical State Line (CSL) which is a stress state of \textit{DPC} yield surface [16, 17]. Since it indicates a division point of consolidation stress state and shear failure stress state for granular materials, CSL provides both of consolidation cap parameters and Drucker-Prager failure parameters. Cap yield surface has been drawn by adding uniaxial compaction stress state that is easily determined by lateral force measurement during closed die compaction. Also, parameters of shear failure line have been identified by adding failure stress state which is measured by simple compression failure test for a green compact. By those examinations, we have succeeded in the estimation of materials parameters for iron powder in the \textit{DPC} model [18] and prediction of failure occurrence in multi-step parts compaction [19]. Results of finite element method (FEM) analysis shows good corresponding with actual die compaction characteristics, but the influence of frictional force during compaction was not considered in the estimation of yield parameters. In this study, frictional force on the die wall in lateral force measurement test has been considered by improving testing device, thus its effect has been eliminated from the estimation of yield parameters. Then the differences in materials parameters on different particle size and different amount of interparticle lubricant have been investigated and its validity on compaction force with a magnitude of the density distribution of green compact has been confirmed by FEM analysis.

2. Definition of Drucker-Prager Cap model

2.1. Yield function

Yield criterion of the \textit{DPC} model in ANSYS is defined by the relationship between the first invariant of stress $I_1$ and second invariant of deviatoric stress $\sigma_2$. The Compaction cap function $Y_c$, Shear failure function $Y_s$ and Expansion cap function are given by equation (1) and (2). Moreover, these functions are combined with equation (3).

$$Y_c = 1 - H(K_o - I_1)\left(\frac{I_1 - K_o}{R_cY_s(K_o, \sigma_o)}\right)^2$$  \hspace{1cm} (1)

$$Y_s = \sigma_o - A\exp(BI_1) - \alpha I_1$$  \hspace{1cm} (2)

$$F(I_1, J_2, K_o, \sigma_o) = J_2' - Y_c(I_1, K_o, \sigma_o)Y_s^2(I_1, \sigma_o) = 0$$  \hspace{1cm} (3)

Where Compaction cap parameter $R_c$ means the ratio of elliptical $I_1$ to $J_2$ axis. $\sigma_o$ is the cohesion related hardening parameter with consolidation. Shear envelope exponential coefficient $A$, Shear envelope exponent $B$ and Shear envelope linear coefficient $\alpha$ are material parameters which define shear yield stress respectively. $H$ and $K_o$ of equation (1) express Heaviside function and a key flag indicating the current transition point at which the compaction cap surface and shear failure surface intersect. Note that the Heaviside function roles as follow;

1. When $I_1$ is greater than $K_o$, the compaction cap takes no effect on yielding. In other words, only shear failure function $Y_s$ is effective.
2. When $I_1$ is less than $K_o$, the yielding may only happen in the compaction cap portion, which is shaped by both the shear failure function $Y_s$ and cap function $Y_c$ as expressed by equation (1). 

Equation (2) means shear failure envelope is simplified as the linear relationship with intercept $\sigma_o$ and inclination $\alpha$ for $A = B = 0$. Therefore, shear yield surface is expressed as a conical shape having a central axis of mean stress $\sigma_m$ in three principal stress space.
2.2. Hardening function
The cap hardening law is defined by describing the evolution of the parameter \( X_o \), which is the current intersection point of the compaction cap and the \( I_1 \) axis. In other words, hardening is expressed by increasing volumetric plastic strain \( \varepsilon_{pv} \) with consolidation proceeding that is defined as:

\[
\varepsilon_{pv} = W_1^c \left[ \exp \{ D_1^c (X_o - X_i) \} - 1 \right]  
\]

where: \( X_i = \) Initial value of compaction cap yield pressure \( X_o \) at which the cap function takes effects, \( W_1^c = \) Maximum possible volumetric plastic strain which is defined from densification curve of the powder material, \( D_1^c = \) Hardening parameter.

2.3. DPC model in \( I_1 - \sqrt{J_2'} \) plane
According to equations (3), the yield surface of the plane DPC model in \( I_1 - \sqrt{J_2'} \) can be drawn schematically as shown in figure 1. Subsequent yield surface can be defined as work hardening behavior which is obtained by variations in density ratio to \( \rho_0 \) from \( \rho_i = \) Initial yield surface by updating initial parameters \( X_i \) and \( \sigma_c \). These parameters are not able to measure directly but can be estimated by interpolation from the stress states of several density ratios.

![Figure 1. Schematic drawing of yield surface of the DPC model](image)

3. Various examinations to identify the parameter of yield surface in the DPC model
3.1. Uniaxial failure test and single shear test
Uniaxial failure test of green compact provides a point of stress state in failure by uniaxial principal stress ( \( \sigma_1, \sigma_2 = \sigma_3 = 0 \) ). The uniaxial failure stress state is obtained as:

\[
\sqrt{J_2'} = \sigma_1 / \sqrt{3}  
\]

\[
I_1 = \sigma_1  
\]

The shape of green compact for uniaxial failure test is cylindrical in 10 mm diameter with 15 mm height.
Single shear test of green compacts in several compact densities provides CSL. In previous work, CSL has identified [15]. When a green compact executes shear failure, vertical displacement on the shear failure plane indicates a positive value which means volume expansion. In the case of vertical force, which exceeds a certain value, vertical displacement indicates a negative value which means consolidation occurrence due to higher mean stress (hydrostatic stress) state. The stress states at which the vertical displacement was kept in zero during shear were estimated by a single shear test. Thus, we can identify $K_o$ directly by the estimation of CSL. The slope of failure lines $\alpha$ and evolution of cohesion $\sigma_o$ with densification proceedings can be estimated by equation (2) by using stress states of CSL and uniaxial failure. The shape of green compact for a single shear test is cylindrical in 20 mm diameter with 8 mm height.

3.2. Lateral force measurement test

Lateral force measurement provides a point of stress state in densification by measuring forming force and lateral force during closed-die compaction. Principle of the lateral force measurement device is shown in figure 2. The device is composed of servo cylinder which gives compaction load as forming force $F_{upper}$ and die set with load cells which measure lateral force $F_L$ and transmitted force $F_{lower}$ respectively. Frictional force occurs on the inner surface of the mold deduces applied forming force, therefore $F_{lower}$ becomes lower than $F_{upper}$ and influence of such frictional force should not be ignored in the identification of compaction cap parameters. The green compact which shape is a square column with 8 mm $\times$ 8 mm sectional dimension can be compacted.

$\sigma_{upper}$ and $\sigma_{lower}$ are derived from $F_{upper}$ and $F_{lower}$. Therefore, $\sigma_z$ is given by:

$$\sigma_z = \sigma_{lower} \left( \frac{\sigma_{upper}}{\sigma_{lower}} \right)^{\frac{Z}{H}}$$

(7)

where: $H$ means the whole height of the powder part in the die, $Z$ = representative value for the height of powder part. In this study, $Z$ was considered as central in the powder. Therefore, $H / Z$ was 1 / 2. So, equation (7) is rewritten as follows:

$$\sigma_z = \sigma_{lower} \left( \frac{\sigma_{upper}}{\sigma_{lower}} \right)^{\frac{1}{2}}$$

(8)
Therefore, by using $\sigma_z$ and lateral pressure $\sigma_L$, the stress state is given by:

\[ I_1 = \sigma_1 + 2\sigma_L \]

\[ \sqrt{J_2} = |\sigma_1 - \sigma_L|/\sqrt{3} \]

where: $\sigma_1 = \sigma_2$, $\sigma_2 = \sigma_3 = \sigma_L$.

3.3. Experimental condition

Iron powder with zinc stearate, which is an additive as interparticle lubricant, has been used. Zinc stearate was spread on the inner surface of the mold in the lateral force measurement test. It is necessary to prepare green compact specimens with proper density for both uniaxial failure test and single shear test. The density ratios $\rho$, which are defined as the ratio of the compact density to material density, were chosen as 0.70, 0.75, 0.80 and 0.85. Atomized iron powders having approximately 100 $\mu$m mean particle size with 1.0 mass % zinc stearate (Fe 100 $\mu$m-1.0 %) and 0.5 mass % zinc stearate (Fe 100 $\mu$m-0.5 %) were tested. Besides, carbonyl iron powder having more fine particles size, which is approximately 10 $\mu$m with 1.0 mass % zinc stearate (Fe 10 $\mu$m-1.0 %) were also tested.

4. Results and discussion

4.1. Critical state line

Figure 3 shows the CSL as a relation between vertical stress $\sigma$ and shear stress $\tau$ on the shear failure plane of powder compacts for several density ratio. It is well known that finer particle size shows fewer consolidation characteristics due to poor flowability, therefore the slope of CSL in 10 $\mu$m particle size becomes higher as it shows higher values of shear strength for each density ratio. Since less amount of interparticle lubricant decreases flow ability, also 0.5 mass% zinc stearate showed a higher slope of CSL.

4.2. Compaction yield characteristics

Figure 4 shows an example of the variations of punch stroke and applied forces in the lateral force measurement test. In loading, each force increases exponentially with increasing punch stroke. The friction coefficient on the inner surface in the loading stage is derived by:

\[ \mu = \frac{F_{upper} - F_{lower}}{F_L} \]

The values of $\mu$ were approximately 0.10 for each tested powder. In the unloading stage, each force decreases almost linearly but lateral force remains significantly. The values of $F_L / F_{upper}$ were 37% for atomized powder and 41% for finer carbonyl powder. It indicates that the finer particle size requires higher stress to densification because of too much point of contact which having frictional resistance.
Figure 3. The critical state line of tested powders

Figure 4. Variations of punch stroke and forces (Fe 100 μm-1.0%)

Figure 5 shows the loading pass in $I_1 - \sqrt{J_2}$ plane for each tested powder. Poor compaction characteristics of Fe 10 μm-1.0% powder is clearly recognized. Interparticle lubricant improves compaction characteristics slightly. However, the influence of $I_1$ to $\sqrt{J_2}$ is comparatively negligible for each tested powder. It is considered that magnitude of work hardening increases significantly in finer particle size but hydrostatic pressure dependence of the deviatoric stress is not varied if substrate materials properties such as elastic modulus and tensile strength are same for the powder.

4.3. Estimation of yield surface of the DPC model

Figure 6 shows an estimated yield locus for each density ratio. The stress states which were investigated by different test, uniaxial failure, single shear and lateral force measurement were combined by DPC models. However, since $\sqrt{J_2}$ values at the critical stress states of Fe 10 μm-1.0% powder are shown at lower than the compaction cap stress states, $K_o$ value is not identified directly by a single shear test. It was considered that the shear failure occurs at different plane due to the clearance which is necessary to conduct single shear in case of finer particle compact. To avoid such crack occurrence at the different
plane with applied vertical force, modification of the shape of the specimen to cube shape having square shear plane will be effective.

(a)Fe $100 \mu m$-$1.0\%$

(b)Fe $10 \mu m$-$1.0\%$

**Figure 6.** Estimated yield surfaces of the DPC model for different particle size

Table 1 shows the yield parameters which have been identified to describe figure 6. The slope of shear failure envelope $\alpha$ was constant in each density ratio, and dependence exists on the particle size difference. It is also influenced by decreasing the amount of lubricant as it becomes 1.5 times higher in case of Fe $100 \mu m$-0.5 % powder. Thus, the interparticle lubricant decreases the shear strength of the compact and its behaviour can be implemented by $\alpha$ in the DPC model. Poor compaction characteristics of Fe $10 \mu m$-1.0% powder is implemented by lower hardening parameter $D_1^c$ with higher compaction cap parameter $R_c$. It indicates that incrementation of densification in hydrostatic pressure rapidly decreases, thus the yield by deviatoric stress state becomes effective relatively in case of finer particle size. In contrast, little difference in $W_1^c$ and $D_1^c$ on amount of interparticle lubricant was recognized.

| Powder          | Fe 100 \( \mu m \)-1.0 % | Fe 10 \( \mu m \)-1.0 % |
|-----------------|---------------------------|---------------------------|
| Density ratio   | 0.50                      | 0.47                      |
|                 | 0.70                      | 0.70                      |
|                 | 0.75                      | 0.75                      |
|                 | 0.80                      | 0.80                      |
| $R_c$           | 3.3                       | 4.0                       |
| $X_0$           | -18                       | -3.6                      |
| $\sigma_1$ [MPa]| 2                         | 19.7                      |
| $\alpha$        | 0.200                     | 0.275                     |
| $W_1^c$         | 0.693                     | 0.755                     |
| $D_1^c$         | 0.0022                    | 0.00142                   |

**Table1.** Yield parameters for the identification of DPC yield surfaces

4.4. **FEM results and validation in densification curve**

Figure 7 shows the relation between applied compaction force and density ratio in comparison of simulation and experimental result measured by lateral force measurement test. Despite measurement error in the single shear test for Fe $10 \mu m$-1.0% powder, FEM results showed good correspondence since its yield locus was complemented by the other two testings with a probable slope of CSL as mentioned in figure 6(b). Insufficient density calculated at the bottom corner of the compact reflects adequately damage occurrence during ejection from mold in actual green compact in fine particle size as the photograph mentioned in figure 7(b). Fe $100 \mu m$-0.5 % powder also shows good correspondence with the experimental result. Thus, it is concluded that proper yield parameters were estimated by the proposed procedure in this study.
5. Conclusions
Critical stress state and uniaxial failure stress state have been determined by a single shear test and uniaxial failure test for green compact for different particle size. Also, compaction cap parameter has been estimated by lateral force measurement test by instrumented die compaction system which can eliminate the influence of frictional force on the inner surface of compaction mold. By those estimations, materials input parameters for the DPC model have been identified successfully, and differences in particle size and amount of lubricant can be recognized adequately. FEM analysis of simple closed-die compaction has been conducted. As a result, the materials input parameters for the DPC model on different powder properties have been successfully identified as the FEM analysis fairly predicts compaction force and density distribution on uniaxial compaction.

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