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Calculated Stress and Strain Conditions of Lubricated Potassium Chloride Powders during Die-Compression

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The stress and strain distributions in a compressed powder bed of potassium chloride lubricated with magnesium stearate were calculated on the basis of the theory of limiting equilibrium states specified by the Mohr criterion for the axially symmetrical problems and of the stress-strain relations. The slopes of yield locus and wall yield locus for the powder bed, which were required for the calculations, were 0.426 and 0.0516, respectively. The calculated results were compared with those for an unlubricated powder bed at P_U (the upper punch pressure) $=10.0$ kg/cm^2. More uniform distributions on the boundary surfaces and within the powder bed appeared in the present lubricated system. The stress distributions became large on the upper and lower punch surfaces as P_U increased. A region of higher stress appeared within the powder bed even at $P_U=10.0$ kg/cm^2, and was clearer at $P_U=60.0$ kg/cm^2.

Keywords——tablets; compression; lubrication; powder; numerical calculation

Introduction

The die compaction of particulate materials is a common operation in the pharmaceutical and related industries. The powder flow in the die is controlled by the internal and die wall frictions which prevent the uniform transmission of the pressure applied to the powder bed and result in a density gradient within the compact, in the die or after ejection from the die.

The patterns of pressure and density distributions have been measured experimentally for many kinds of powders.[1,2] Macleord *et al.*[2] examined the effect on these patterns of several factors that affected the die wall friction. Their results showed that a substantial gradient appeared within the compact in the system without die wall lubricant, and that uniformly dense compacts were obtained in the system containing internal lubricant.

Fukumori *et al.*[3] discussed the stress distribution in the compact, which could be calculated on the basis of the theory of limiting equilibrium states specified by the Mohr–Coulomb criterion and the stress-strain relations proposed by them.

In the previous papers,[4] it was found that a lubricant (magnesium stearate) admixed with potassium chloride powders affected the frictional conditions on the die wall and made the density distributions on the punch surfaces more uniform. These results indicate that the stress and strain distributions within the powder bed in the lubricated system must be very different from those in the unlubricated system. It is of interest to study the differences in these distributions. Such investigations should provide useful information on the compaction and lubrication of pharmaceutical powders.

In this paper, the stress and strain distributions in a lubricated powder bed are calculated according to the methods described by Fukumori *et al.*[3a] A system of potassium chloride powders lubricated with 2.0% magnesium stearate was chosen for calculation. The mechanical properties of the system required for calculation have already been obtained.[4b] The results are compared with those for the unlubricated system. The effect of upper punch pressure on the distributions is discussed.

Theoretical and Calculations

1. Theoretical

The theories described in detail by Fukumori *et al.*,[3a] on which the present calculations are based, are briefly described here.

It is assumed that the compressed powder bed in the die is in the limiting equilibrium state. Thus, the equilibrium equations for axially symmetrical problems and the Mohr–Coulomb criterion are applied to this compressed powder system. The boundary conditions for the stress on the surface of the upper punch are set by assigning arbitrary values to the coefficients (*i.e.*, a_i and b_i) of the following polynomials:

$$\sigma_z = \sum_{i=1}^{m} a_i \left(\frac{r}{r_w}\right)^{i-1}, \quad \phi = \sum_{i=1}^{n} b_i \left(\frac{r}{r_w}\right)^{i-1} \qquad \text{Eq. 1}$$

where σ_z is the stress normal to the punch surface and ϕ is the angle of the z-axis to the maximum principal stress direction. Then, the stress field within the powder bed can be calculated. On the other hand, according to the incremental theory, the theory of infinitesimal strain may be applied to this system. The total increment of the strain is the sum of the increments produced by the one-dimensional compression and shearing. By dividing the powder bed into small finite elements, the displacement at any point can be calculated. The boundary conditions for the displacement on the surface of the lower punch can be obtained as follows:

$$dU_r = 0, \quad dU_z = const. \qquad \text{Eq. 2}$$

where dU_r and dU_z are the components of the increment of displacement in the r- and z-directions, respectively. The coefficients of the polynomials can be corrected by minimizing the sum of the squares of deviation from these boundary conditions.

2. Numerical Values

For potassium chloride powders lubricated with 2.0% magnesium stearate, all numerical values required for calculation are presented in the previous paper.[4b] The wall friction was determined by sliding the compacts on the wall. The kinetic friction, corresponding to the minimum value during sliding, was used. The slope of the wall yield locus was 0.0516. The maximum and minimum principal stresses were determined by measuring the lateral pressure within the die, which was divided into three parts. The slope of the yield locus was 0.426. The compression tests in the die (diameter: 20.11 mm) with a single punch were carried out repeatedly at four weight levels (3.92, 7.84, 15.67 and 23.51 g). The extrapolation of the compression data to zero weight gave the stress-strain relation for one-dimensional compression. This relation and the above mentioned values of two loci were used in numerical calculation in the present study.

The calculations were carried out on the compression in a cylindrical die from one side by the upper punch. The cylindrical co-ordinate system (r, θ, z) was used (see Fig. 5), and the origin was set on the upper punch. At the beginning of compression, when the radius of the die(R) is assumed to be 10.0, the corresponding height of the powder bed(H) was calculated to be 20.97 in the present system.[4b] This means that the lower punch surface is located at z=20.97.

Results and Discussion

1. Calculated Stress Conditions on the Boundary Surfaces

The calculated distributions of stress normal to the upper and lower punches (σ_z) are shown in Figs. 1 and 2, respectively. The ratios of σ_z to P_U (the upper punch pressure) and

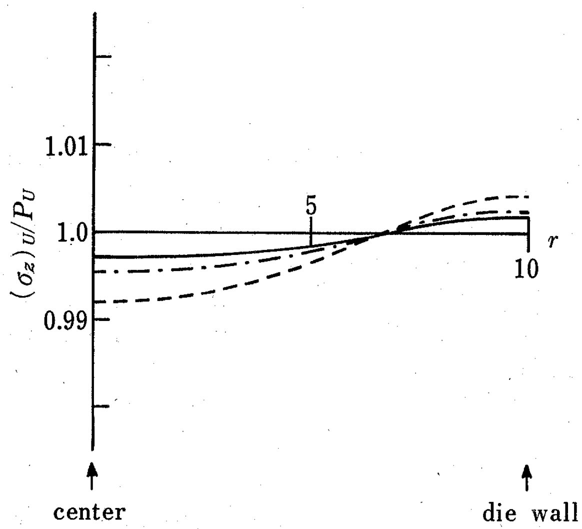

Fig. 1. Calculated Distributions of Stress Normal to the Upper Punch

P_U: ——— 10.0, —·—·— 30.0, ··········· 60.0 kg/cm².
$(\sigma_z)_U/P_U$ the ratio of the stress to the upper punch pressure.
r: the radial distance from the center.

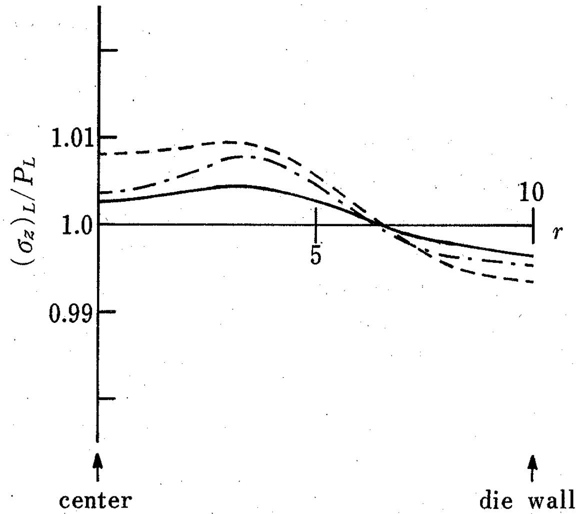

Fig. 2. Calculated Distributions of Stress Normal to the Lower Punch

P_U: ——— 10.0, —·—·— 30.0, ··········· 60.0 kg/cm².
$(\sigma_z)_L/P_L$: the ratio of the stress to the lower punch pressure.
r: the radial distance from the center.

to P_L (the lower punch pressure) are plotted as the ordinate and the radial (*r*-axial) distance from the center is plotted as the abscissa. These distribution curves are similar to those obtained experimentally[4-6]: on the upper punch, the normal stress was low around the center and increased near the die wall, while on the lower punch, this relation was reversed.

For $P_U=10.0$ kg/cm², the calculated curves in the lubricated system are compared with those in the unlubricated system.[3b] As expected, a more uniform distribution was obtained in the present (lubricated) system. In the unlubricated system, the changes in σ_z/P_U or σ_z/P_L were very large (in the ranges of 0.95 to 1.05 on the upper punch and 0.90 to 1.15 on the lower punch). In the present system, these values are within 1% of P_U or P_L. As shown in Fig. 1, in the region near the die wall the stress on the upper punch dose not increase as sharply as in other calculated[3] and measured[4-6] results. The small slopes in this region are probably due to the low value of the coefficient of wall friction.

Strijbos *et al.*[5] measured the boundary stress conditions of the powder bed in the lubricated and/or unlubricated die with concentrically split punches. They detected marked differences between these conditions. They also found that the effect of lubrication on the stress conditions was greater on the lower punch than on the upper punch. These two findings support the calculated results in the present study. However, the lubrication effect on the stress conditions on both punches is greater than that obtained by Strijbos *et al.* It should be noted that lubrication adopted in this study is different from the die wall lubrication used by them.

When the distribution curves at three levels of P_U are compared with each other, the higher P_U is, the greater the slope of the curve is on both punch surfaces. The three curves intersect at a point where *r* is about 7.0 and the ratio of stress is about 1.0. The latter finding is in accord with the experimental results[6] for unlubricated potassium chloride powders at much higher levels of P_U.

Fig. 3 shows the calculated distributions of stress normal to the die wall (σ_r) for $P_U=10.0$ kg/cm², where the ratio of σ_r to P_U and the *z*-axial distance from the upper punch surface are plotted as the abscissa and ordinate, respectively. The values of σ_r/P_U are much higher than the values (0.2 to 0.4) obtained in the unlubricated system.[3a] The value of σ_r decreases almost linearly toward the lower punch. However, the curve of σ_r, as well as the curve of σ_z, is more uniform than in the unlubricated system. The higher value of σ_r and its uniformity

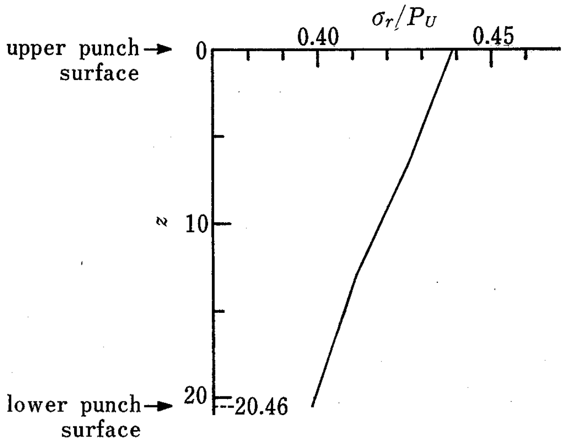

Fig. 3. Calculated Distributions of Stress Normal to the Die Wall

P_U: 10.0 kg/cm².
σ_r/P_U: the ratio of the stress to the upper punch pressure.
z: the z-axial distance from the upper punch surface.

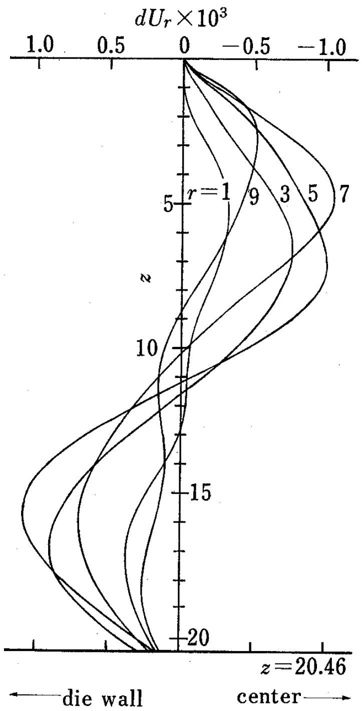

Fig. 4. Calculated Increments of Displacement in the r-axial Direction when P_U was changed from 8.0 to 10.0 kg/cm²

dU_r: the component of the increments of displacement in the r-axial direction.
z: the z-axial distance from upper punch surface of the nodes.
r: the r-axial distance from the center of the nodes.

may be due to the decrease in internal friction caused by the admixed lubricant. Strijbos *et al.*[5] reported similar results using the die wall lubricant. It may be considered that the changes in σ_r observed in this work are due to decreases in both internal and die wall frictions. For the other levels of P_U, the ratio of σ_r/P_U and its distribution were nearly the same as for $P_U = 10.0$ kg/cm².

2. Calculated Conditions within a Powder Bed

The increments of displacement in the r-axial direction (dU_r), are plotted in Fig. 4, when P_U was changed from 8.0 to 10.0 kg/cm². These patterns are nearly the same as those obtained in the unlubricated system.[3b] In the upper part of the bed, the displacement occurs toward the center. In the lower part, this occurs toward the die wall. However, the values of dU_r at the nodes in the present system are less than one-third of those in the unlubricated system.

The stress distribution within a lubricated powder bed was calculated for $P_U = 10.0$ kg/cm² (Fig. 5). The lines shown in the figure are the isobars of the normal component of stress acting on the failure surface (σ_Y). The lines are more gently sloped than those in the unlubricated system, in which the lines vary rapidly, especially near the die wall.[3b] Also, the range of σ_Y is narrower. The difference between the maximum and minimum values is less than 10% of the maximum, indicating that the distribution of σ_Y is nearly uniform throughout the bed. These gently-sloping curves may be due to the low value of the coefficient of die wall friction produced by the admixed lubricant, that is, lower resistance on the die wall. Even at $P_U = 10.0$ kg/cm², a region having a higher value of σ_Y than its surroundings can be detected near $z = 12$ on the z-axis. This result in the lubricated system is almost the same as that in the unlubricated system where such a region appeared on the z-axis at the position of about $0.6H$ from the upper punch surface.[1b,3]

Macleord *et al.*[2] and Train[1] examined the density distribution within the powder bed with or without internal or die wall lubricant. When the lubricant was applied to powders or the die, more uniformly dense compacts were obtained.

The stress distribution within the lubricated powder bed for $P_U = 60.0$ kg/cm² is also

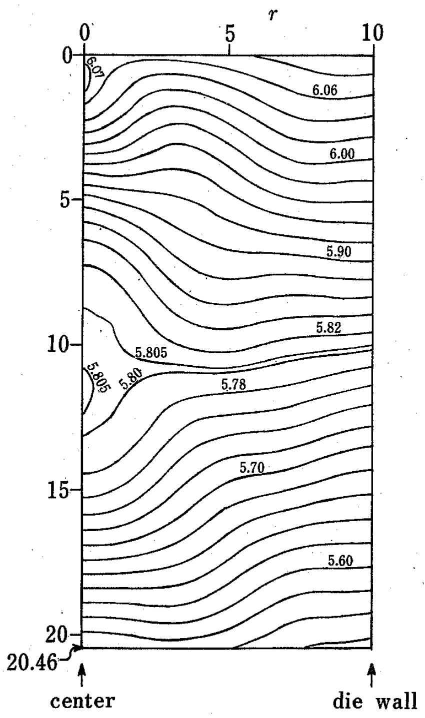

Fig. 5. Calculated Distribution of Stress acting normally on the Failure Surface within a Lubricated Powder Bed

P_U: 10.0 kg/cm².
R(r co-ordinate at the die wall): 10.0.
H(z co-ordinate on the lower punch surface): 20.46.

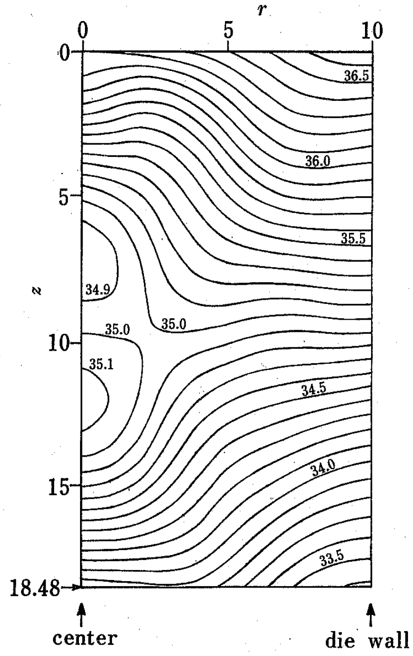

Fig. 6. Calculated Distribution of Stress acting normally on the Failure Surface within a Lubricated Powder Bed

P_U: 60.0 kg/cm².
R(r co-ordinate at the die wall): 10.0.
H(z co-ordinate on the lower punch surface): 18.48.

shown in Fig. 6. The isobars are also gently sloping, as in the case of P_U=10.0 kg/cm² (Fig. 5). The region having the higher value of σ_Y is clear and broad. The change in the distribution pattern with increasing P_U is in accordance with that reported by Train.[1b] On the other hand, there is little difference between the positions at the two levels of P_U.

Conclusions

The boundary stress conditions and the stress and displacement distributions within the powder bed were evaluated for a system of potassium chloride powders lubricated with magnesium stearate.

At the stage when P_U=10.0 kg/cm², the calculated results were compared with those in the unlubricated system obtained previously. More uniform patterns were found in the present lubricated system both on the boundary surfaces and within the bed. However, the region having the higher value of σ_Y within the bed was apparent on the z-axis.

In addition, the calculated results for several levels of P_U (10.0, 30.0 and 60.0 kg/cm²) were compared. The slopes of the distribution curves of normal stress on the upper and lower punch surfaces were greater with increasing P_U. The region having the higher value of σ_Y was detected more clearly at P_U=60.0 kg/cm².

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