Introduction of a New Index for the Prediction of Capping Tendency of Tablets

Ken-ichi SUGIMORI,*[,a] Shoichi MORI[a] and Yoshiaki KAWASHIMA[b]

Technology Development Laboratories, Takeda Chemical Industries, Ltd.,[a] 2-17-85, Juso-honmachi, Yodogawa, Osaka 532, Japan and Gifu Pharmaceutical University,[b] 5-6-1, Mitahora-higashi, Gifu 502, Japan. *Received July 26, 1988*

Capping within a tablet was quantitatively estimated by using the capping ratio, defined as the ratio of the tablet strength to that of the uniformly compacted tablet.

Capping index was also newly defined as the ratio of the extrapolated residual die wall pressure to the binding strength of compacted powder. It was found that capping occurred when the capping index exceeded unity. This fact indicated that capping can be regarded as cracking within a tablet caused by high residual die wall pressure. The requirements to achieve uniform compaction were clarified from these results.

It was found that the extrapolated residual die wall pressures of convex-faced tablets were related to those of flat-faced ones. It was also found that tablets consisting of more elastic powders had a lower residual die wall pressure.

Keywords tablet; tablet strength; capping; capping ratio; capping index; die wall pressure

Introduction

When pharmaceutical powders are compacted into tablets, cracking, usually called capping, sometimes occurs in the tablets. To overcome this problem, many studies have been done from practical and theoretical viewpoints.[1,2] The characteristics of die wall pressure during the compaction process have been investigated,[3-7] and attempts have been made to express quantitatively the capping tendency by the use of defined indices.[8-11] However, the mechanism of capping is not fully understood, and no method for quantitative estimation of cracking within a tablet has yet been established.

In this paper, the authors propose the capping ratio as a quantitative measure of capping, and also newly define the capping index as the ratio of the extrapolated residual die wall pressure to the binding strength of compacted powders. The usefulness of this index for the prediction of capping is discussed, and the mechanism of capping was clarified. Furthermore, the mechanism of the uniform compaction of powders was elucidated, and the relationship between the residual die wall pressure and tablet shape or size is discussed.

Experimental

Materials Various powders with different capping tendencies were used. The powders without capping tendency were caffeine (Shizuoka Caffeine Industries, Ltd., Japan) and cornstarch (Japan Cornstarch Co., Ltd., Japan). The powders with capping tendency were ethoxybenzamide (ethenzamide, Yoshitomi Pharmaceutical Industries, Ltd., Japan), lactose (DMV, Holland), and hydroxybutylphenetidin (bucetin, Yamato Chemicals Co., Ltd., Japan). A mixture of lactose and cornstarch (LCM) (mixing ratio=2:1) was used as a reference powder without capping tendency, and granules composed of ethenzamide and bucetin (EBG) were used as a powder having only a slight capping tendency. These granules (EBG) were prepared by mixing 45% ethenzamide, 45% bucetin and 10% α-starch (Nichiden Chemicals Co., Ltd., Japan), wet massing, drying in a vacuum dryer at 40°C, crushing, and sieving through a 500 μm screen.

Tablet Preparation Powders were compacted into tablets using a compacting test apparatus (Autograph, Shimadzu Seisakusho Co., Ltd., Japan) with a die of 8.5 mm internal diameter and either flat-faced punches or concave-faced ones of 6.5 mm radius of curvature. The compacting conditions were as follows: 200 mg of powder, 2000 kg/cm^2 compaction pressure, 5 mm/min compression speed, 0.5 mm/min decompression speed, and 100 mm/min ejection speed. Before each compaction, the die was lubricated with a very small amount of magnesium stearate (Sakai Chemical Industries, Ltd., Japan).

Apparatus for Uniform Compaction To enable the quantitative estimation of capping, it was necessary to obtain tablets with no cracks. In this paper the loaded-ejection-type compacting apparatus was employed to prepare these tablets. This apparatus was originally designed by Funakoshi *et al.*[12,13]

The structure of this apparatus is illustrated in Fig. 1. It was used combined with the compacting test apparatus described above. On this apparatus, powder was compressed and decompressed in the usual manner. However, when the axial pressure decreased to about 100 kg/cm^2 in the decompression process, the spring, which was set on the lower punch holder, began to exert pressure, and the tablet in the die was ejected under the axial pressure applied. After ejection, the axial pressure was removed. Any type of powder could be compacted into uniform tablets without capping by using this apparatus. The conditions of this compaction were the same as those of the tablet preparation described above.

Measurement of Tablet Strength Tablet strength was measured by the tablet hardness tester (Erweka, Germany). Several tablets were compressed in a diametrical direction and the crushing strength was measured. These values were then averaged.

Measurement of Die Wall Pressure The die wall pressure during the compaction process was measured using a specially designed die, the center section being thinned to allow for easy measurement. The die wall pressure was detected with a strain gauge attached to the outer surface of the die, and measured by a strain meter equipped with a recorder. The output values were calibrated with paraffin wax and silicon rubber. The conditions of compaction were the same as those of the tablets preparation.

Binding Strength of Compacted Powders To measure the binding strength of the compacted powders, cylindrical compacts were prepared using the loaded-ejection-type compacting apparatus described above. The compaction was carried out using a die of 11.3 mm internal diameter and flat-faced punches. The compacting conditions were as follows: 1000 mg of powder, 2000 kg/cm^2 compaction pressure, 0.5 mm/min compression and decompression speed, and 25 mm/min ejection speed. Before compaction, the die was lubricated with a very small amount of magnesium stearate. The thicknesses of these compacts were about 7—8.5 mm.

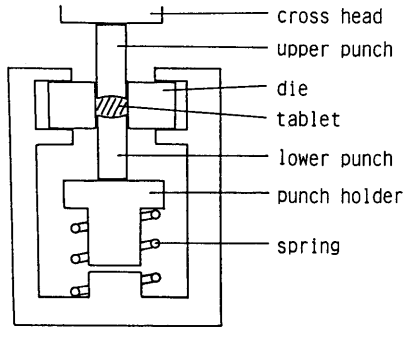

Fig. 1. Loaded-Ejection-Type Compacting Apparatus

These cylindrical compacts were subjected to a fracturing test by compressing them in an axial direction by using the compacting test apparatus.

The compression speed in the fracturing test was 0.5 mm/min. In this test, the true value of the strain was obtained by subtracting the blank strain from the total strain because the apparatus was strained to a small extent.

Results and Discussion

Quantitative Evaluation of Capping In the analysis of the capping phenomenon, the most difficult problem is that there is no established method to quantitatively estimate capping.

Usually, a friability test is used for the measurement of capping. In this test, tablets are struck in a rotating friabilator, and the number of tablets fractured after the rotation are counted. However, these data are directly affected by the rotating conditions, and the physical meaning of the data is not clear.

Funakoshi reported a method to measure the internal strength distribution in a tablet by boring a tablet with a small drill.[13] Kigasawa *et al.* analyzed the internal strength distribution in a tablet using a similar tester.[14-16] Even if these methods are used, it is difficult to quantitatively estimate capping.

In this study, a method for the quantitative estimation of capping was established by using the loaded-ejection-type compacting apparatus which allows compaction of all types of powders without capping.[12,13] A tablet with the same size as the test tablet was prepared as a standard tablet under the same condition by using the loaded ejection method. Then the strength of the tablet prepared by the ordinary method (F) was compared with that of this standard tablet (F_u). The capping ratio was defined in this study as follows:

$$\text{capping ratio} = (F_u - F)/F_u \quad (1)$$

For the tablet without capping, F is equal to F_u, and the capping ratio becomes zero. For the tablet with complete capping, F tends towards zero and the capping ratio will increase towards unity. When a tablet cracks slightly, the capping ratio takes a value between zero and unity. Thus, the capping of a tablet can be quantitatively expressed by the defined capping ratio.

The tablets were prepared by using the 8.5 mm internal diameter die and either flat-faced punches or concave-faced ones of 6.5 mm radius of curvature at 2000 kg/cm^2 compaction pressure, and their capping ratios were determined. The results are tabulated in Tables I and II.

It is empirically known that ethenzamide, lactose and bucetin tend to crack and convex-faced tablets or thinner tablets crack more easily than flat-faced ones or thicker ones. The resultant capping ratios in Tables I and II explain quantitatively this empirical information. From these results, it was concluded that the capping ratio is a valid index to estimate capping quantitatively.

TABLE I. Capping Ratio and Residual Die Wall Pressure of a Flat-Faced Tablet

Material	Weight (mg)	Thickness (mm)	Strength F (kg)	Strength F_u (kg)	Capping ratio $(F_u-F)/F_u$	Residual pressure (kg/cm^2)
Caffeine	200	2.58	12.2	12.3	0.01	95
LCM	200	2.53	4.5	5.0	0.10	90
Cornstarch	200	2.60	3.4	4.0	0.15	5
EBG	200	2.95	9.3	10.9	0.15	220
Ethenzamide	200	2.98	11.3	15.0	0.25	240
Lactose	200	2.53	1.7	2.9	0.41	195
Bucetin	200	3.10	0.8	1.5	0.47	165

Residual Die Wall Pressure and Capping Ratio Figures 2—5 show the die wall pressures during the decompression process, where the powders were compacted at 2000 kg/cm^2 using the 8.5 mm internal diameter die and either flat-faced punches or concave-faced ones of 6.5 mm radius of curvature. The flat-faced tablets without capping tendency (Fig. 2) had considerably lower residual die wall pressures than

TABLE II. Capping Ratio and Residual Die Wall Pressure of a Convex-Faced Tablet

Material	Weight (mg)	Thickness (mm)	Strength F (kg)	Strength F_u (kg)	Capping ratio $(F_u-F)/F_u$	Residual pressure (kg/cm^2)
Caffeine	200	3.94	13.9	14.3	0.03	230
LCM	200	4.00	5.2	5.5	0.05	225
Cornstarch	200	4.07	2.2	2.6	0.15	10
EBG	200	4.40	7.9	11.3	0.30	440
EBG	250	5.11	11.3	12.7	0.11	360
EBG	300	5.85	12.4	13.4	0.07	320
Ethenzamide	200	4.47	5.5	9.8	0.43	455
Lactose	200	4.05	0	3.7	1.00	420
Bucetin	200	4.53	0	1.7	1.00	295

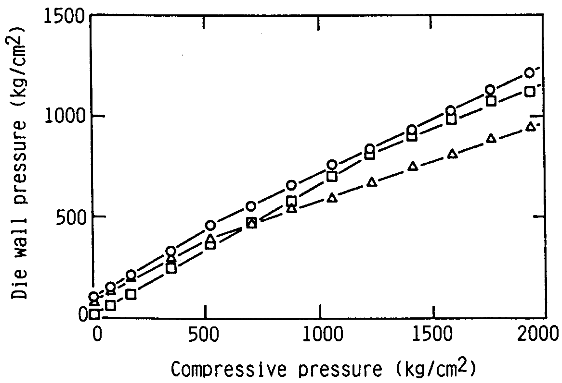

Fig. 2. Die Wall Pressure of a Flat-Faced Tablet during the Decompression Process

○, caffeine; △, LCM; □, cornstarch.

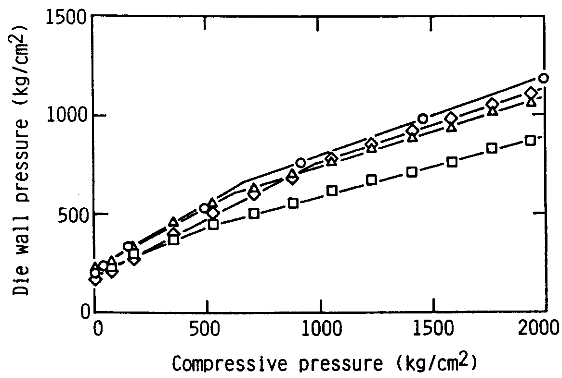

Fig. 3. Die Wall Pressure of a Flat-Faced Tablet during the Decompression Process

○, EBG; △, ethenzamide; □, lactose; ◇, bucetin.

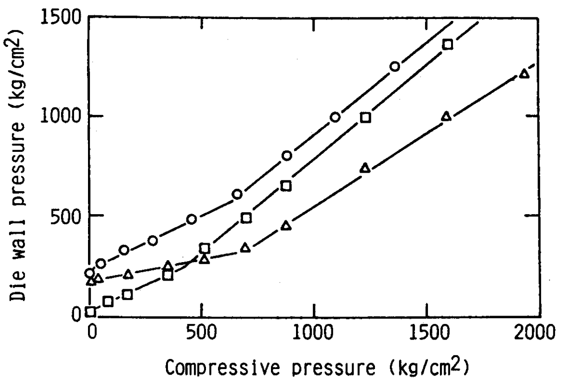

Fig. 4. Die Wall Pressure of a Convex-Faced Tablet during the Decompression Process

○, caffeine; △, LCM; □, cornstarch.

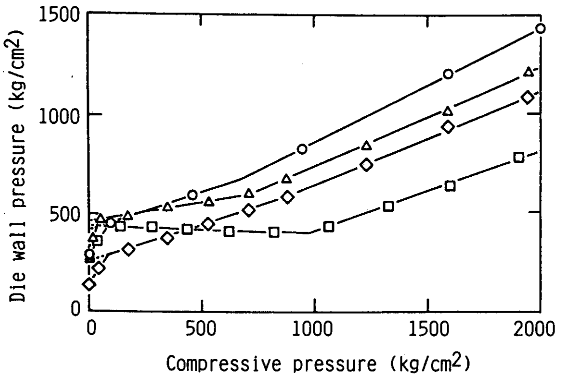

Fig. 5. Die Wall Pressure of a Convex-Faced Tablet during the Decompression Process

○, EBG; △, ethenzamide; □, lactose; ◇, bucetin.

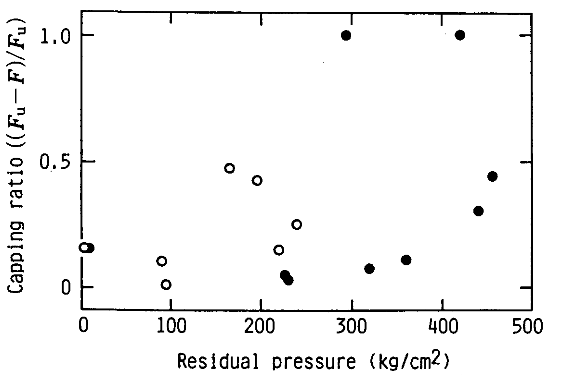

Fig. 6. Relationship between Residual Die Wall Pressure and Capping Ratio

○, flat-face; ●, convex-face (6.5R).

those with capping tendency (Fig. 3). The convex-faced tablets (Figs. 4 and 5) had higher residual die was pressures than the flat-faced tablets (Figs. 2 and 3), and a drastic drop in the die wall pressure at the final stage of the decompression process was found (Fig. 5) for the convex-faced tablets with capping tendency.

The extrapolated residual die wall pressures were determined from Figs. 2—5, and are summarized in Tables I and II. These values were compared with the capping ratios in the same tables, and their relationship is illustrated in Fig. 6. No correlation was found between the extrapolated residual die wall pressure and the capping ratio. This indicated that there are some other parameters involved in the capping process than the residual die wall pressure.

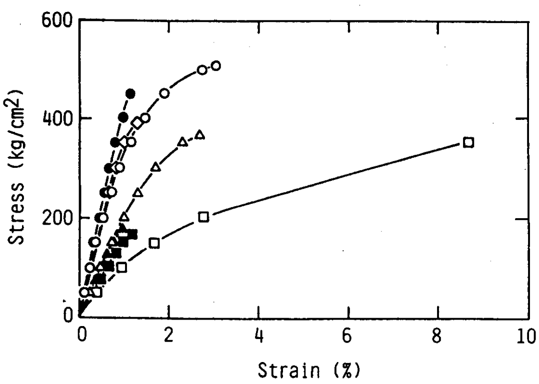

Fig. 7. Stress-Strain Curves of Cylindrical Compacts

○, caffeine; △, LCM; □, cornstarch; ◇, EBG; ●, ethenzamide; ▲, lactose; ■, bucetin.

Table III. Crushing Strength of a Cylindrical Compact

Material	Compact length (mm)	Crushing strength (kg/cm^2)	Maximum strain (%)
Caffeine	7.06	505	3.11
LCM	7.11	365	2.68
Cornstarch	7.34	350	8.68
EBG	8.24	390	1.32
Ethenzamide	8.44	450	1.16
Lactose	7.18	175	0.97
Bucetin	8.49	165	1.19

Table IV. Size Effect on the Crushing Strength of a Cylindrical Compact

Weight (mg)	Diameter (mm)	Length (mm)	Crushing strength (kg/cm^2)	Maximum strain (%)
1000	11.3	8.16	432	1.29
800	8.5	11.52	422	1.23
600	8.5	8.64	449	1.32
400	8.5	5.75	458	1.23

Capping Index and Capping Ratio The strength of a tablet is usually measured by compressing the tablet in a diametrical direction using a tablet hardness tester. However, this strength is tensile strength.[17] It is very difficult to measure the radial crushing strength of a tablet.

In this work, the powders were compacted into uniform cylindrical compacts using the loaded-ejection-type compacting apparatus. These compacts were compressed axially and crushed. The stress-strain curves are shown in Fig. 7. This crushing strength was regarded as the binding strength of the compacted powders and the values are listed in Table III.

The axial crushing strengths of variously sized cylindrical compacts are shown in Table IV, where it was found that the size effect on the strength was sufficiently small. The values in Table IV differ slightly from those of EBG in Table III. This was due to the difference in the mixing lots of powders.

The strength of the compacts in Table III was compared with the capping ratios in Tables I and II. No correlation was observed, however, between these values.

It was considered that the relationship between the extrapolated residual die wall pressure (Q_r, as a fracturing force) and the binding strength of compacted powders (P_c, as a resistance force) was significant. The ratio of these two values was newly defined as a capping index (Eq. 2).

$$\text{capping index} = Q_r/P_c \quad (2)$$

The binding strength of compacted powder is considered to be identical in both flat-faced and convex-faced tablets. Therefore the same value of P_c was used for the calculation of the capping index for flat-faced and convex-faced tablets.

The capping indices calculated from the data in Tables I, II and III were compared with the capping ratios in Tables I and II. The correlation between these values is illustrated in Fig. 8.

A good correlation was observed between the capping index and the capping ratio. That is, it was found that no capping occurred when the capping index was smaller than unity ($Q_r < P_c$), whereas capping occurred slightly when the index was approximately equal to unity ($Q_r = P_c$), and occurred extensively when the index exceeded unity ($Q_r > P_c$). Thus the capping index defined in Eq. 2 was concluded to be a useful parameter for the quantitative prediction of capping.

These results indicated that capping took place when the residual die wall pressure exceeded the binding strength of the compacted powders, and proved that capping can be regarded as the cracking of the compact brought about by high residual die wall pressure.

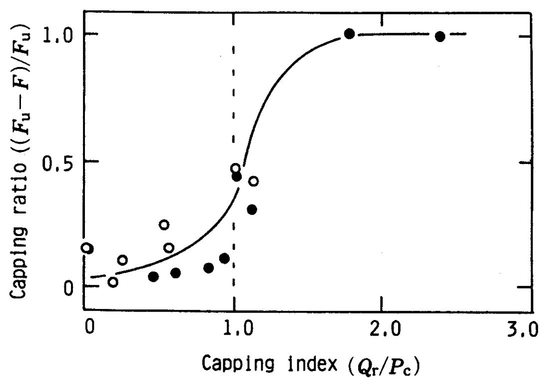

Fig. 8. Relationship between Capping Index and Capping Ratio
○, flat-face; ●, convex-face (6.5R)

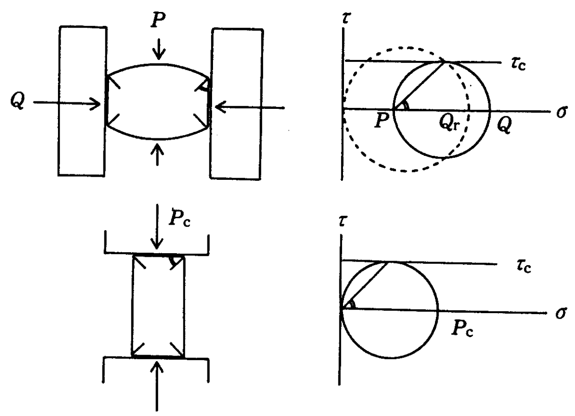

Fig. 9. Stress and Mohr's Circle for a Tablet (Upper) and for a Cylindrical Compact (Lower)

Mechanism of Capping The stresses in the tablet and Mohr's circle are schematically shown in Fig. 9 (upper-part). The tablet undergoes a low axial pressure and a high radial pressure during the final stage of the decompression process. The shear stresses in various directions within the tablet are expressed by Mohr's circle which passes through (P,0) and (Q,0). The shear stress is the highest in the direction of 45° and its value equals $(Q-P)/2$. it is considered that, when the shear stress overcomes the shear strength of the compacted powders (τ_c), the tablet will crack in the direction shown in the figure.

The stresses and Mohr's circle in the crushing test of a cylindrical compact are also shown in Fig. 9 (lower part). The shear stress in the compact is the highest in the direction of 45° and the compact cracks in the direction illustrated in the figure. Thus the shear strength of the compact (τ_c) is regarded as $P_c/2$, the same as that of the tablet.

The Mohr's circle of the extrapolated residual die wall pressure (Q_r) is represented by the dotted circle which passes through (0, 0) and (Q_r, 0). To compare the extrapolated residual die wall pressure with the binding strength of a compact means to compare this dotted Mohr's circle with the Mohr's circle of the crushing test. If the capping index is larger than unity ($Q_r > P_c$), the dotted Mohr's circle of residual pressure becomes larger than that of the crushing test, and then the tablet will crack.

If the powders are compacted into tablets at high speed, the relaxation of the stress will be reduced, resulting in higher residual die wall pressure and the tablets will have a greater tendency to cap.

Effect of Loaded Ejection As described above, any type of powder can be compacted into variously shaped tablets without capping by the use of the loaded-ejection-type compacting apparatus. This apparatus is characterized by tablet ejection under low applied axial pressure (*ca.* 100 kg/cm^2).

Figure 10 shows the stresses which act within the tablet during the decompression process, where P is compressive stress, Q is die wall stress and τ is maximum shear stress produced by P and Q, and equals $|(P-Q)/2|$.

In the ordinary decompression process, τ decreases, reaches zero and increses again. Eventually τ reaches the shear strength of the compact (τ_c) as illustrated with a

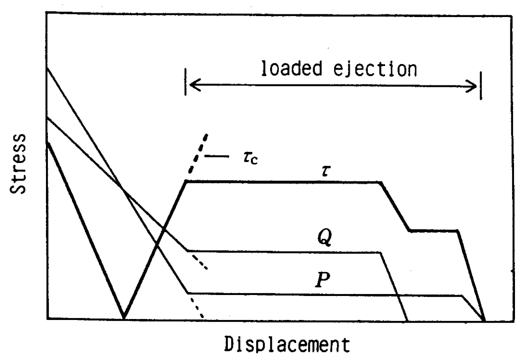

Fig. 10. Loaded Ejection Effect; Stress within a Tablet during the Decompression Process

TABLE V. Relationship between Residual Die Wall Pressure and Tablet Thickness

Material	$A=(Q_{rc}-Q_{rf})/Q_{rf}$	$B=(T_v-T_e)/T_e$	A/B
Caffeine	1.42	2.13	0.67
LCM	1.50	1.98	0.75
Cornstarch	1.00	1.83	(0.55)
EBG	1.00	1.34	0.75
EBG	0.64	0.85	0.75
EBG	0.45	0.62	0.73
Ethenzamide	0.90	1.27	0.70
Lactose	1.15	1.87	0.61
Bucetin	0.79	1.21	0.65
Average	—	—	0.7

dotted line in the figure, and then capping will occur.

In the case of the loaded ejection method, the tablet is ejected under low applied axial pressure, and P, Q and τ show the curves indicated with solid lines in the figure. The tablet is decompressed and ejected from the die with the shear stress lower than the shear strength (τ_c), where capping never occurs.

Characteristics of Extrapolated Residual Die Wall Pressure The characteristics of extrapolated residual die wall pressure, an important factor to determine capping, were investigated. The extrapolated residual die wall pressures of convex-faced tablets (Q_{rc}) were higher than those of flat-faced ones (Q_{rf}) as shown in Tables I and II, but were not proportional to the reciprocal of the contact area with the die wall. The relationship between these two residual pressures is discussed below.

Table V indicates $(Q_{rc}-Q_{rf})/Q_{rf}$ $(=A)$, $(T_v-T_e)/T_e$ $(=B)$ and the ratio of these values (A/B), where T_v is the thickness of the flat-faced tablet having the same diameter and the same volume as the convex-faced tablet, and T_e is the edge thickness of the convex-faced tablet. It was found that the ratio (A/B) was approximately 0.7 irrespective of the kind of powder. The ratio of cornstarch was lower than the others. However, this was considered to be due to experimental error because the residual die wall pressure of cornstarch was very low.

These results indicated that the shape or size effect on residual die wall pressure was independent of the type of powder. Thus the following empirical equation was obtained from Table V.

$$(Q_{rc}-Q_{rf})/Q_{rf}=0.7\cdot(T_v-T_e)/T_e \quad (3)$$

The extrapolated residual die wall pressure of variously sized convex-faced tablets can be calculated from that of the flat-faced tablet by using this equation, and consequently the capping of these tablets can be predicted.

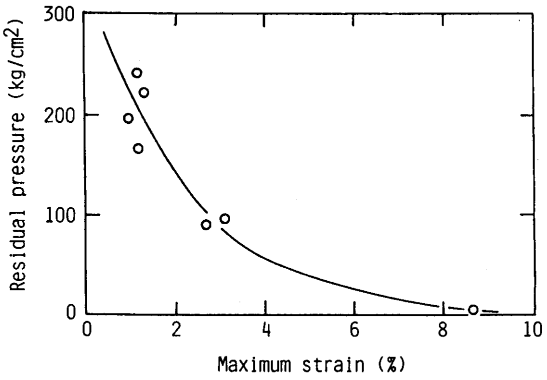

Fig. 11. Relationship between Maximum Strain of a Cylindrical Compact and Residual Die Wall Pressure of a Flat-Faced Tablet

The extrapolated residual die wall pressures of the flat-faced tablets were related to the maximum strain of the cylindrical compacts on the crushing test shown in Fig. 7. The relationship between these two values is presented in Fig. 11. A good correlation was found between these two values. The extrapolated residual die wall pressure decreased as maximum strain increased. This implied that the residual die wall pressure depended on the extent of elastic deformation of the compacted powder, and that the powders with high elasticity showed low residual die wall pressure.

References

1) D. Train, *J. Pharm. Pharmacol.*, **8**, 745 (1956).
2) E. Shotton and D. Ganderton, *J. Pharm. Pharmacol.*, **13**, 144T (1961).
3) W. M. Long, *Powder Metallurgy*, **6**, 73 (1960).
4) S. Leigh, J. E. Carless and R. W. Burt, *J. Pharm. Sci.*, **56**, 888 (1967).
5) E. Shotton and B. A. Obiorah, *J. Pharm. Sci.*, **64**, 1213 (1975).
6) E. Doelker and E. Shotton, *J. Pharm. Pharmacol.*, **29**, 193 (1977).
7) E. N. Hiestand, J. E. Wells, C. B. Peot and J. F. Ochs, *J. Pharm. Sci.*, **66**, 510 (1977).
8) P. J. Jarosz and E. L. Parrott, *J. Pharm. Sci.*, **71**, 607 (1982).
9) A. B. Bangudu and N. Pilpel, *J. Pharm. Pharmacol.*, **37**, 289 (1985).
10) O. Ejiofor, S. Esezobo and N. Pilpel, *J. Pharm. Pharmacol.*, **38**, 1 (1986).
11) W. E. Jetzer, *J. Pharm. Pharmacol.*, **38**, 254 (1986).
12) Y. Funakoshi, T. Kajiura and T. Asogawa, *Zairyo*, **18**, 89 (1969).
13) Y. Funakoshi, *Zairyo*, **24**, 673 (1975).
14) K. Kigasawa, H. Iimura, H. Sugimura and A. Tanizaki, *Yakugaku Zasshi*, **94**, 1281 (1974).
15) K. Kigasawa, H. Iimura, H. Sugimura and A. Tanizaki, *Yakugaku Zasshi*, **94**, 1286 (1974).
16) K. Kigasawa, H. Iimura, H. Sugimura, A. Tanizaki, H. Sugimura and H. Shimizu, *Yakugaku Zasshi*, **95**, 796 (1975).
17) J. T. Fell and J. M. Newton, *J. Pharm. Sci.*, **59**, 688 (1970).