

Physics of Tablet Compression XIV

Lateral Die Wall Pressure During and After Compression

By **TAKERU HIGUCHI, TSUGIO SHIMAMOTO, STUART P. ERIKSEN,**
and **TAKATSUKA YASHIKI***

Measurements of pressure exerted by various substances on the die wall during and after compression of tablets have shown that the information derived can be related directly to the ease of formation and ejection of these tablets. The side wall pressure has been found to decay at a measurable rate after the normal compressional force is abruptly removed. Similar studies also have been carried out on preformed tablets subjected to repeated compression.

IN EARLIER publications (1, 2), devoted to studies on the physics of tablet compression, a method for direct measurements of lateral forces transmitted to the die wall and the relative magnitude of such forces developed during formation of tablets from a number of organic and inorganic substances were reported. Although these results could not be totally rationalized at the time, the data suggested that more information concerning pressure transmitting characteristics during and after compression would be highly desirable in understanding the compressional process.

In the present paper, the following studies were carried out: (a) the relation between the exerted normal pressure and the lateral pressure during compression, (b) the decay of residual side wall pressure, (c) the correlation between the residual side wall pressure and the required force to eject the formed tablet, and (d) the pressure transmitting characteristics when the formed tablet was compressed repeatedly. The results obtained from these measurements suggest that studies of this type yield direct information concerning the ease with which any given charge can be formed into a tablet and the force which may be required to remove the tablet from the die.

EXPERIMENTAL

Reagents.—All materials compressed were of purest grade commercially available and were dried at a suitable temperature. The individual charges were weighed to the nearest milligram necessary to yield a 5-mm. tablet and stored in desiccators prior to use. Table I lists the various materials compressed.

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* Present address: Takeda Chemical Industries, Ltd., Osaka, Japan.

Apparatus.—The general procedure and instruments employed in this study have been described earlier in some detail (2). Tablets were compressed with a high-force hydraulic press equipped with two $\frac{3}{8}$ -in. flat-faced punches and a corresponding die. Upper punch pressure was transmitted directly to the loading button of a Baldwin load cell. Two strain gauges were cemented on the die wall—one normal and the other parallel to the die bore, forming two arms of a Wheatstone bridge circuit. The electrical responses of the load cell on the upper punch and of the strain gauges on the die wall were recorded simultaneously on a Sanborn dual-track magnetic oscillograph, as previously described (2, 3). The die wall was coated with a small amount of hydrocarbon lubricant prior to use to minimize the friction between the particle and the die wall. Tablet hardness was determined by using a Strong-Cobb tablet hardness tester (4).

Procedure.—A known weight of material adjusted to provide a tablet of a constant final thickness of 5 mm. was introduced into the die. The recorder was turned on, and the compression process was carried out smoothly over approximately 30 seconds. Unless indicated otherwise, compressions were to a pressure of 18,300 p.s.i. on the upper punch. At the instant maximum pressure was reached, as read on the oscillograph, the upper punch was released. The recording of the die wall response was continued for a few minutes after the compression of tablet was completed.

TABLE I.—MATERIALS COMPRESSED INTO TABLETS

Substance	Wt. of Die Fill, mg.	Remarks
Sodium chloride	705	20–40 mesh
Potassium chloride	685	20–40 mesh
Potassium bromide	960	20–40 mesh ^a
Potassium carbonate	650	40–60 mesh
DASC ^b	490	Smaller than 60 mesh
Sucrose	495	20–40 mesh
Lactose	470	Smaller than 60 mesh
Sulfathiazole	490	Smaller than 60 mesh
Phenacetin	410	40–60 mesh
Aspirin	475	Smaller than 60 mesh
Acetanilid	412	Smaller than 60 mesh
Phenobarbital	433	Smaller than 60 mesh
Stearic acid	360	Smaller than 60 mesh
Talc	830	Smaller than 60 mesh
Magnesium stearate	350	Smaller than 60 mesh

^a Unless indicated otherwise. ^b Dihydroxy aluminum sodium carbonate.

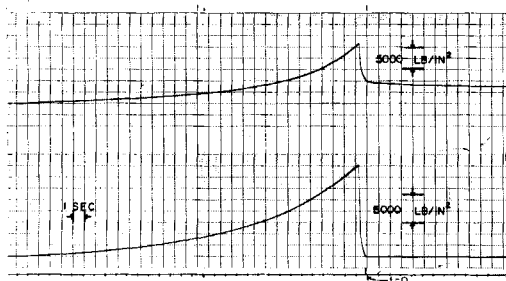


Fig. 1.—Record of upper punch and die wall pressure for compression of sodium chloride. Key: upper half, pressure-time record for the die wall; lower half, pressure-time record for the upper punch; bottom scale, time scale.

The formed tablet was compressed again without removal from the die to the same upper punch pressure used initially. The compression cycle was repeated a total of five times; the compressed tablet in the die was ejected by the upper punch at the end of the fifth compression. The required force for ejection was read from the height of the level portion on the upper punch force-time curve, as previously described (3, 5).

RESULTS AND DISCUSSION

Type and Nature of Data Obtainable.—Typical oscillographic records of upper punch force and die wall force are shown in Fig. 1. The lower half of the chart is the force-time record for the upper punch, and the upper half of the chart is that for the die wall. The recording in Fig. 1 was made by punching 705 mg. of sodium chloride crystals. The direct relationship between applied upper punch pressure and pressure transmitted to the die wall during compression of tablet can be obtained by comparing the two curves, as previously described (2); the result for the above example is shown as the lower line in Fig. 2. Although the side wall pressure appears to decrease abruptly as soon as the upper punch is removed from the formed tablet, a substantial portion of the lateral force remains after completion of the compressional process and return

of the normal force to zero. The magnitude of this residual force exerted by the finished tablet on the side wall and its change with time can be read readily from the recorder chart. The side wall pressure appears to decay gradually with time, reaching equilibrium within roughly 2 minutes. The time scale involved certainly would be of concern during production of tablets under commercial conditions.

If the formed tablet like the one produced in the example cited above is repeatedly recompressed without removal from the die, the side wall-normal force relationship appears to be markedly different from that observed during the initial process. The data obtained during repeated recompression of sodium chloride are shown in Fig. 2. It is evident that the response curves of the third, fourth, and fifth compression were much the same as that of the second compression. If the compressed material acted simply as a hydraulic fluid, the lateral pressure would have been equal to the upper punch pressure, and no differences between the first and the subsequent compressions would have been observed. On the other hand, if the formed tablets were ideally rigid and had no deformation tendency, no lateral pressure should have been transmitted to the die wall by subsequent compressions.

The difference in the transmitted die wall pressure between the first and the fifth compression has been plotted as a function of applied upper punch pressure in Fig. 3. The primary difference in die wall pressure response between the initial and final runs is, as expected, at the lowest compressional forces; the difference decreases rapidly with increasing pressure and becomes essentially zero and finally negative at the highest pressures. Although in most cases the general shapes of the curves were similar to that of sodium chloride, there were significant differences.

In Fig. 4, the decay observed with time of the residual die wall pressure immediately following the removal of the compressional force is shown in terms of the logarithm of the difference between the residual die wall pressure at any time, R , and the residual die wall pressure at equilibrium, $R_{eq.}$, for each compression of sodium chloride. The curves obtained do not seem to fit a singular pattern and indicate that the observed over-all rate of decay is

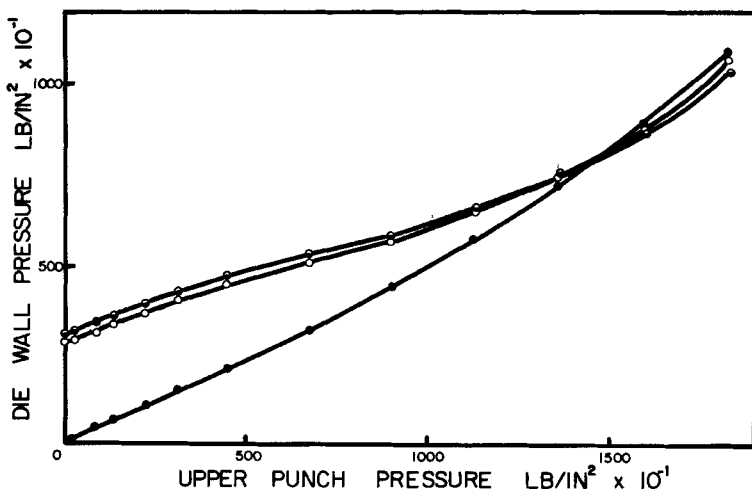


Fig. 2.—Die wall pressure response curves as a function of applied upper punch pressure for sodium chloride. Key: ●, first compression; ○, second compression; ◐, fifth compression.

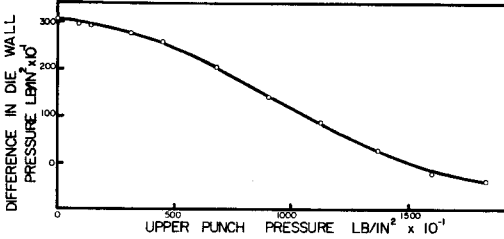


Fig. 3.—The difference in die wall pressure between the first and the fifth compression as a function of applied upper punch pressure for sodium chloride.

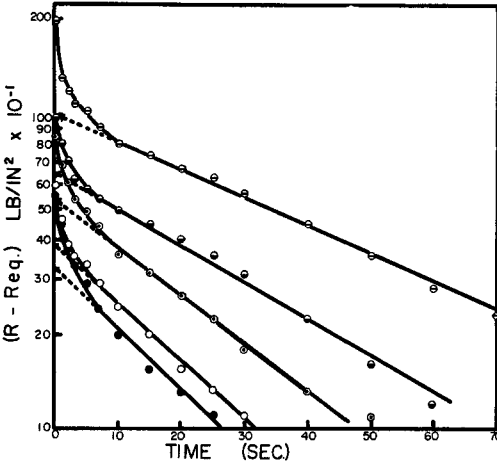


Fig. 4.—The decay curves of residual die wall pressure for sodium chloride. Key: \ominus , first compression; \bullet , second compression; \circ , third compression; \circ , fourth compression; \bullet , fifth compression.

likely a summation of rates of several processes. The changes in the transmitted die wall pressure can probably be attributed to changes in at least two factors—a largely elastic component which is quickly released by elastic deformation after the upper punch is removed from the formed tablet, and a rheologically controlled component which decays slowly through a flow process.

It is apparent on this basis that during the compressional step a considerable amount of mechanical energy becomes stored in the tablet structure. This compressional energy can be divided into several components: (a) the elastic component which is released rapidly and reversibly to the upper punch during its removal. (b) That energy which is dissipated through a relatively slow rheological process in which the side wall pressure produces a flow opposite to that occurring during compression. (c) Residual elastic compressional energy resulting from the residual side wall pressure remaining after cessation of the flow process. This component is relieved on ejection of the tablet. (d) Stress energy arising from internal strain produced by compression and remaining within the formed tablet after its ejection.

It is evident that the data obtainable from these side wall pressure studies can be quite useful in understanding the second and the third items above particularly.

The flow phenomenon, as reflected in Fig. 4, appears to occur in two stages which can be approximated by a relatively single model which assumes that the residual stress is relieved by two noncompeting first-order processes. Even a cursory inspection of the figure suggests that both the initial rapid change and the slower secondary phase occur at fairly reproducible first-order rates for the several compression cycles, despite the fact that the absolute magnitude of the side wall pressure changes significantly with recompression. Speculation regarding the nature of the physical processes which correspond to the rate observation would at this stage be some-

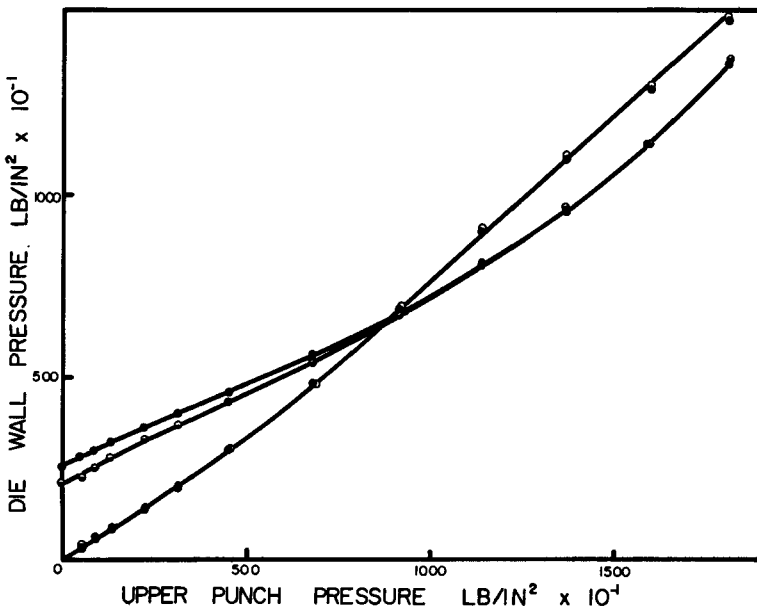


Fig. 5.—Upper punch-die wall response curves for fractionated potassium bromide. Key: \bullet , first compression; \bullet , fifth compression. Both 20-40-mesh particles. \circ , First compression; \circ , fifth compression. Both smaller than 60 mesh.

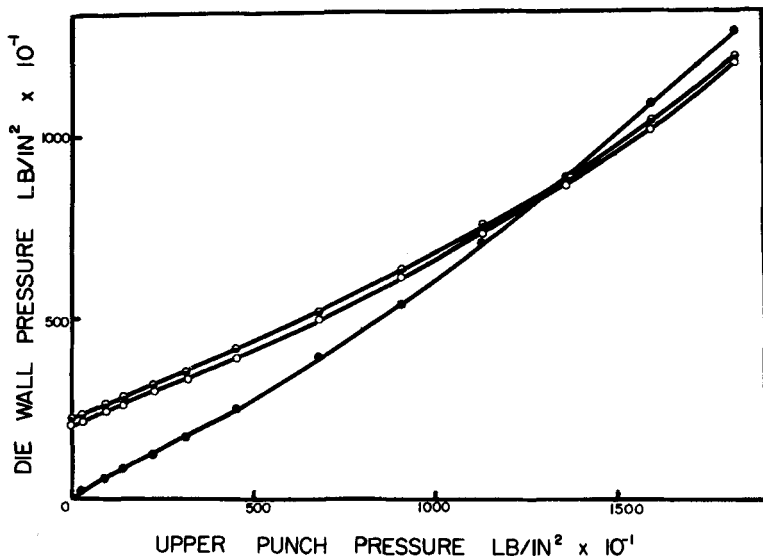


Fig. 6.—Die wall pressure response curves as a function of applied upper punch pressure for potassium chloride. Key: ●, first compression; ○, second compression; ●, fifth compression.

what premature. Nevertheless, it is apparent that these effects reflect in many ways the tablet characteristics which determine their strength, cohesiveness, and ease of production.

Effect of Particle Size.—Changes in particle size of the tested materials appear to have only a minor effect on the pressure transmitting characteristics. In sodium chloride (through a 20-mesh and retained on a 40-mesh screen, through a 40-mesh and retained on a 60-mesh screen, and smaller than 60-mesh), the curves obtained were identical for each sample, and no significant differences were observed between them within experimental variation. For potassium bromide, the behavior of particles smaller than 60-mesh was slightly different from that of coarser particles. In Fig. 5, the curves obtained for two particle size ranges of potassium bromide (through a 20-mesh and retained on a 40-mesh screen and the particles smaller than 60-mesh) are compressed. There was no appreciable difference in pressure transmitting characteristics between the first compression of coarser particles and that of finer par-

ticles. But the fifth compression curve of finer particles showed somewhat higher side wall pressure than for the coarser particles.

Side Wall Pressure Response of a Number of Different Pure Compounds.—Figures 6–11 show experimentally found relationships between the applied upper punch pressure and the pressure transmitted to the die wall for a variety of organic and inorganic compounds. In general, as previously found, the lateral pressure transmitted to the die wall was substantially less than the applied upper punch pressure, apparently because these systems had relatively poor flow tendency.

In an earlier study (2), the die wall–upper punch pressure response curves for alkali metal halides showed a sigmoidal pattern. In the present investigation, the shape of the curves for the first compression of sodium chloride, potassium chloride, and potassium bromide was observed to be somewhat simpler. (See Figs. 2, 5, and 6.) The response curves of subsequent compression cycles for the same system show, however, a sigmoidal tendency with a pronounced plateau region. This difference may be ascribed to small changes in the manner with which the samples are packed into the die.

The results obtained during compression of crystalline stearic acid, acetanilid, and phenacetin were also reported in a previous paper (2). The essentially zero initial slope was found to be common to all three compounds in the earlier study, indicating almost no lateral pressure transmission in the beginning of compression. Each of these three compounds was in the form of platelike crystals. In the present study, powdered forms of these three compounds were compressed. As shown in Figs. 9–11, no marked difference is observed in the shape of curves between these three compounds and others. The response curves show the lateral force developing during the initial phase of compression.

The ratios of maximum die wall pressure to maximum upper punch pressure for the first compression seem to reflect the hardness of crystals employed. The hardest (lactose) have the lowest values and the softest (stearic acid) have the highest values, as shown in the first column in Table III.

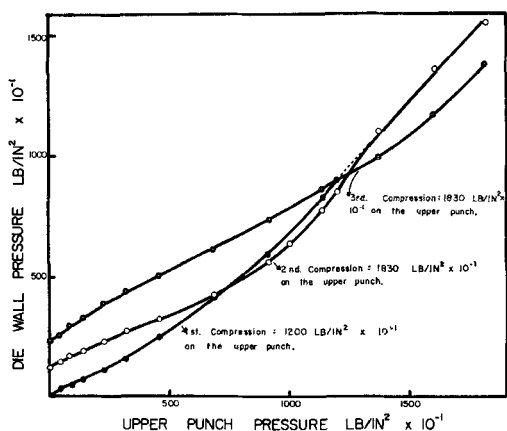


Fig. 7.—Upper punch–die wall response curves for potassium bromide. The first compression is to 12,000 lb./in.² on the upper punch; the others are to 18,300 lb./in.².

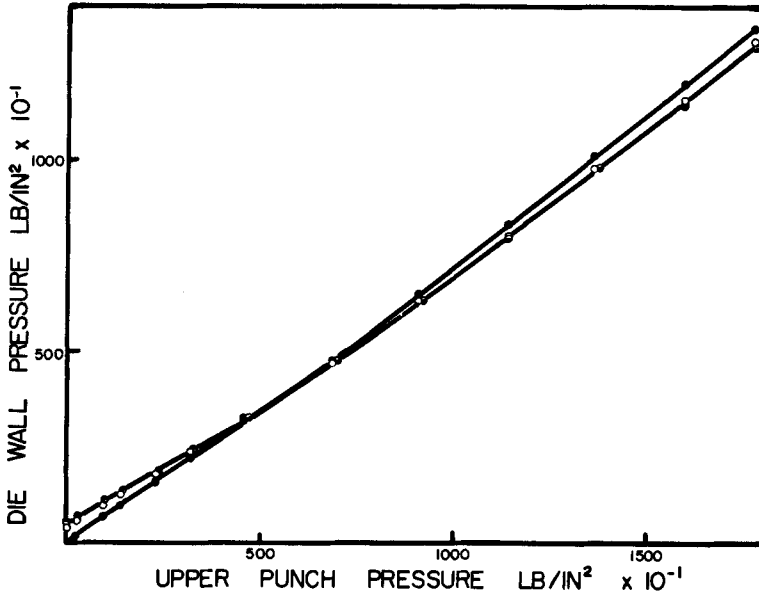


Fig. 8.—Die wall pressure response curves as a function of applied upper punch pressure for aspirin. Key: ●, first compression; ○, second compression; ◐, fifth compression.

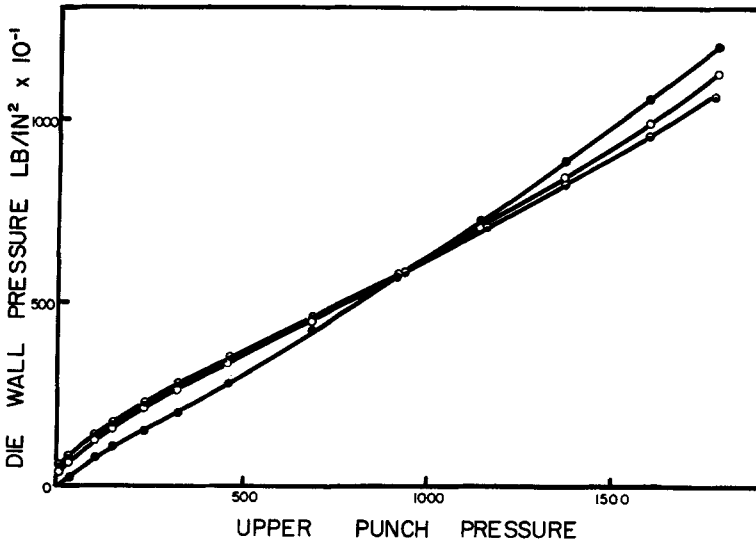


Fig. 9.—Die wall pressure response curves as a function of applied upper punch pressure for phenacetin. Key: ●, first compression; ○, second compression; ◐, fifth compression.

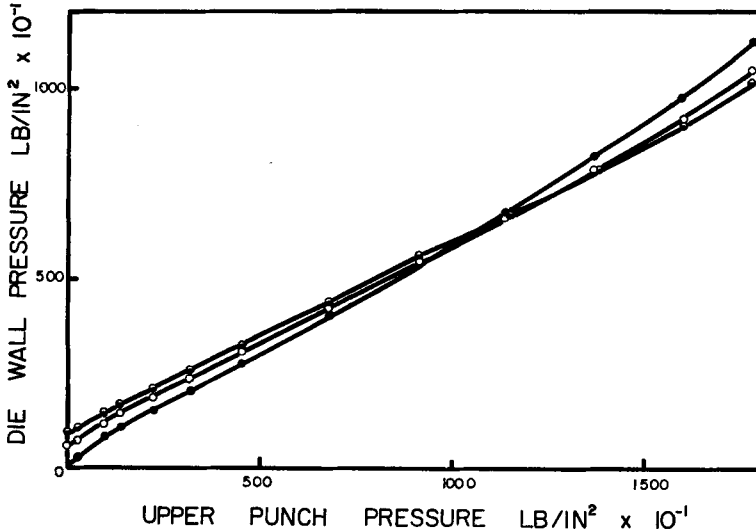


Fig. 10.—Die wall pressure response curves as a function of applied upper punch pressure for acetanilid. Key: ●, first compression; ○, second compression; ◐, fifth compression.

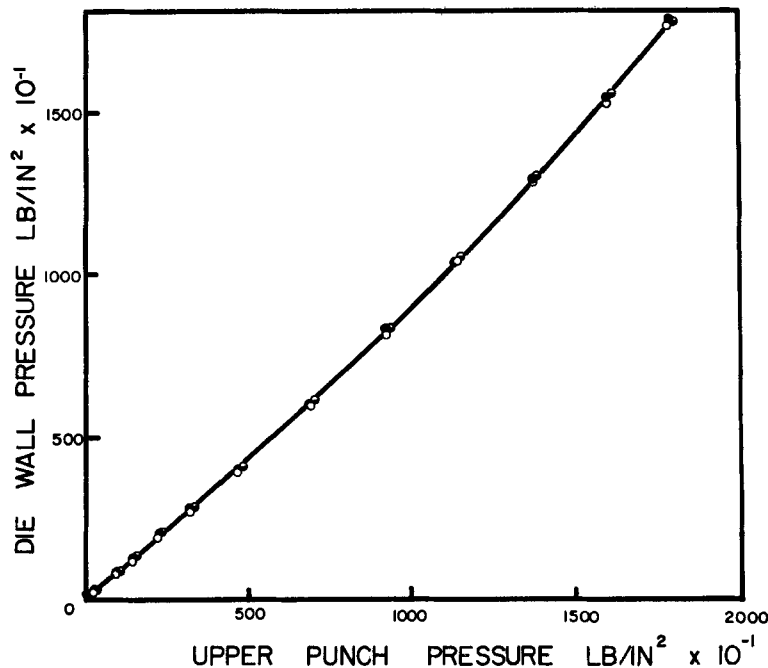


Fig. 11.—Die wall pressure response curves as a function of applied upper punch pressure for commercial stearic acid. Key: \circ , first compression; \bullet , second compression; \odot , fifth compression.

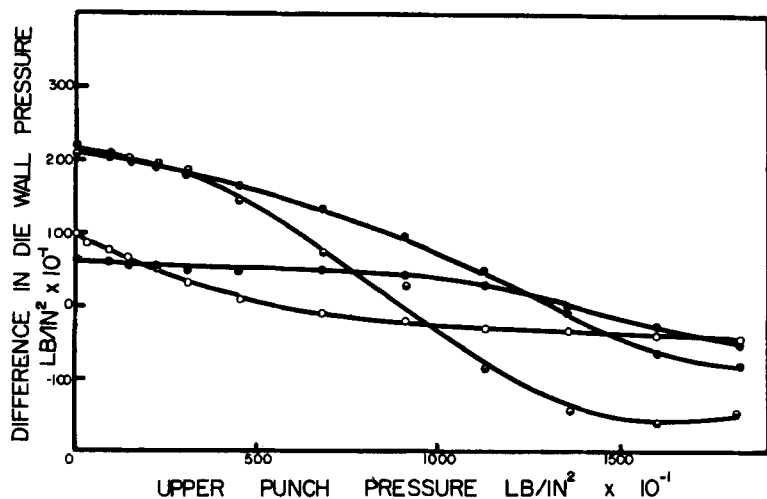


Fig. 12.—The difference in die wall pressure between the first and the fifth compression as a function of applied upper punch pressure. Key: \odot , potassium bromide; \odot , potassium chloride; \circ , DASC (dihydroxy aluminum sodium carbonate); \bullet , potassium carbonate.

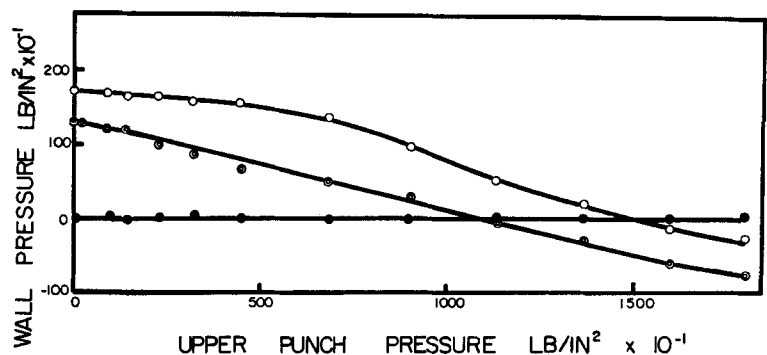


Fig. 13.—The difference in die wall pressure between the first and the fifth compression as a function of applied upper punch pressure. Key: \circ , sucrose; \odot , lactose; \bullet , stearic acid.

Differences in die wall pressure observed during compression between the first and the fifth compression for these same systems have been plotted in Figs. 12-14. The shapes of the curves are much the same as that of sodium chloride, and the differences become below zero at the highest upper punch pres-

sure, indicating that the magnitude of die wall pressure for the fifth compression is lower than that for the first compression. The pressure transmitting characteristic curves for the first and the fifth compression cross at a certain upper punch pressure. (See Figs. 5-10.)

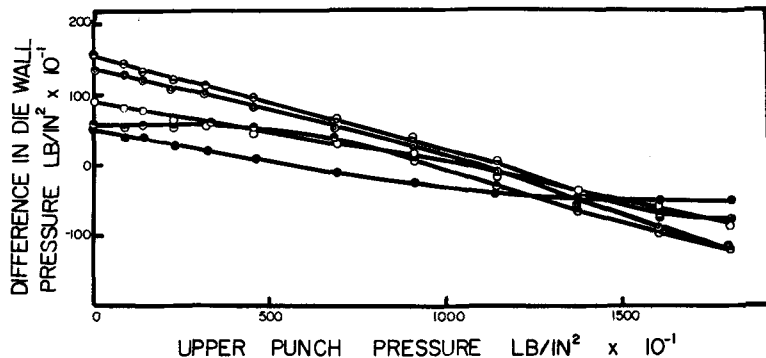


Fig. 14.—The difference in die wall pressure between the first and the fifth compression as a function of applied upper punch pressure. Key: ●, aspirin; ○, acetanilid; ⊕, phenacetin; ⊖, phenobarbital; ⊙, sulfathiazole.

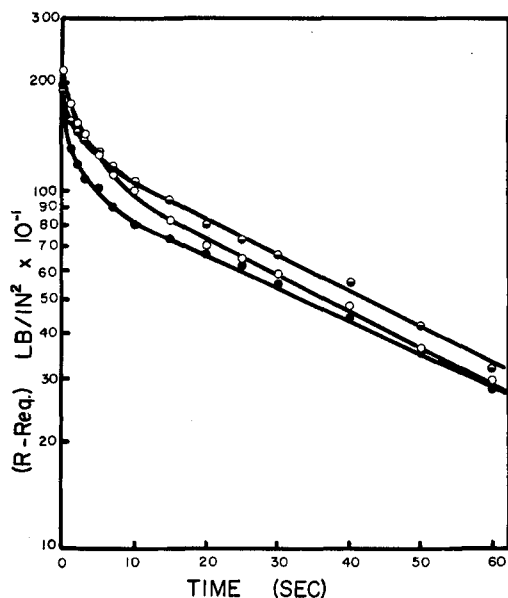


Fig. 15.—The decay curves of residual die wall pressure. Key: ●, sodium chloride; ○, potassium chloride; ⊖, potassium bromide.

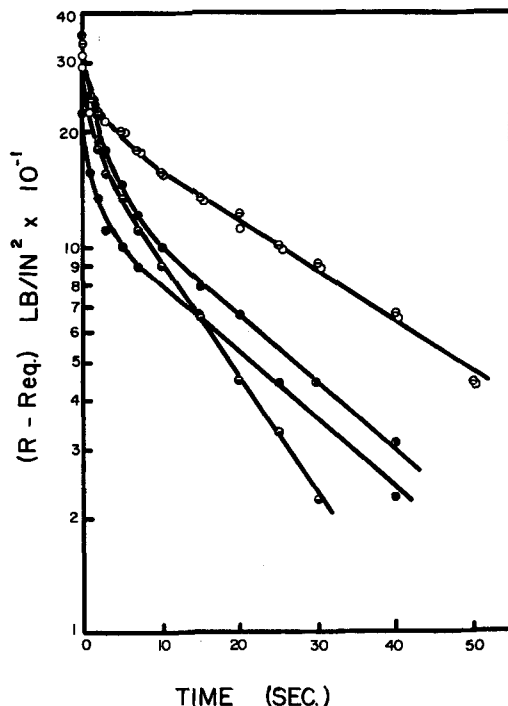


Fig. 16.—The decay curves of residual die wall pressure. Key: ⊙, sucrose; ⊖, lactose; ●, aspirin; ⊖, potassium bromide + 5% magnesium stearate; ○, potassium chloride + 5% magnesium stearate.

In Figs. 15 and 16 the decay observed with time of the residual die wall pressure after the first compression of various substances, as previously mentioned, is shown. The rate of decay may depend on crystal hardness and particle-particle friction. Table II shows that the slopes of the linear portion of the curves plotted in Figs. 15 and 16 appear to follow the certain pattern. The hardest crystals (lactose) have the highest slopes; the softest (alkali metal halides) have the lowest slopes, although some exceptions appear to exist. The slopes also seem to be dependent on particle-particle friction. Alkali metal halides containing 5% by weight of magnesium stearate show higher slopes than the pure crystals. Although these responses are probably dependent to some extent on the rate of compression of each sample, the major effect of variation in the rate of formation on the decay pattern will probably be manifested within milliseconds after release of the compressional force. Thus, they are not of concern in the present instance.

Relationship Between the Ejection Force and Residual Die Wall Pressure.—The ejection forces observed for tested materials are listed in the seventh

TABLE II.—SLOPE OF THE DECAY OF RESIDUAL DIE WALL PRESSURE^a

Substance	Sec. ⁻¹
Sulfathiazole	0.016
Phenacetin	0.016
Lactose	0.030
Sucrose	0.018
Aspirin	0.017
Sodium chloride	0.009
Potassium chloride	0.010
Potassium bromide	0.010
Potassium chloride + 5% magnesium stearate	0.015
Potassium bromide + 5% magnesium stearate	0.015

^a $\Delta \log (R - R_{eq.}) / \Delta t$.

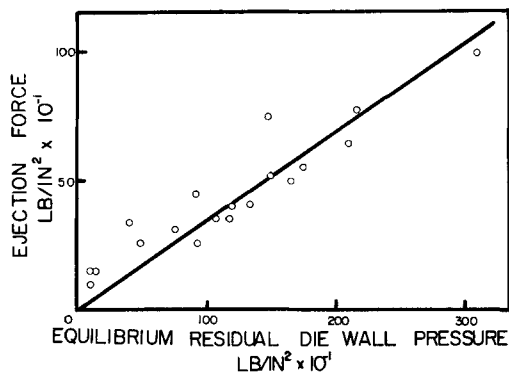


Fig. 17.—Relationship between the ejection force and the equilibrium residual die wall pressure after the fifth compression.

Although some deviations are noticed in Fig. 17, it appears that the two are related directly and linearly. Since the degree of lubrication employed was essentially the same in all cases, this relationship is not totally unexpected. It is apparent that the residual die wall pressure plays an important role in understanding the amount of work required to eject the finished tablets.

CONCLUSION

The die wall pressure decreases rapidly after the upper punch is removed from the formed tablet, but a part of the pressure remains on the die wall after compression.

The decay observed with time of the residual die wall pressure can be approximated by a model based on two noncompeting first-order processes. The rate of decay appears to be dependent on crystal hardness and particle-particle friction.

TABLE III.—DATA OBTAINED FROM THE COMPRESSION OF VARIOUS MATERIALS^a

Substance	Ratio of Max. D.W.P./ Max. U.P.P. (%)	R.D.W.P. ^c (lb./in. ² × 10 ⁻¹)				Hardness ^f Reading	Ejection Force (lb./ in. ² × 10 ⁻¹)
		The First		The Fifth			
		R ₀ ^d	R _{eq.} ^e	R ₀	R _{eq.}		
Sodium chloride	59	436	284	306	311	>26	100
Potassium chloride	71	427	212	382	218	20	78
Potassium bromide	81	410	191	345	210	>26	64
Potassium carbonate	48	80	67	84	77	12	32
DASC ^g	38	142	107	116	107	14	36
Sucrose	49	200	169	191	178	11	55
Lactose	49	156	138	142	133	10	41
Sulfathiazole	52	185	126	196	150	26	52
Phenacetin	66	82	36	93	49	— ^h	27
Aspirin	73	51	33	76	40	13	37
Acetanilid	61	115	58	124	89	3	45
Phenobarbital	60	170	136	151	148	3	76
Stearic acid	96	25	13	25	13	10	15
Talc	65	15	8	20	9	4	15
Magnesium stearate	73	13	8	20	10	6	9
Potassium bromide +2% magnesium stearate	82	173	115	167	115	8	34
Potassium bromide +5% magnesium stearate	85	124	95	120	93	6	27
Potassium chloride +2% magnesium stearate	80	253	167	231	164	10	50
Potassium chloride +5% magnesium stearate	84	191	131	165	120	10	41

^a All compressions were made to a pressure of 18,300 lb./in.² on the upper punch. ^b For the first compression, D.W.P. (die wall pressure) and U.P.P. (upper punch pressure). ^c R.D.W.P., residual die wall pressure. ^d R₀, initial residual die wall pressure. ^e R_{eq.}, equilibrium residual die wall pressure. ^f Using the Strong-Cobb hardness tester. ^g DASC, dihydroxy aluminum sodium carbonate. ^h Acceptable tablets could not be obtained.

column in Table III. The data show that alkali metal halides are associated with high residual die wall pressure and correspondingly high ejection force; the ejection of tablets often is associated with audio accompaniment. On the other hand, magnesium stearate, talc, stearic acid, and phenacetin show low residual die wall pressure, and the ejection forces are correspondingly low. The nature of the relationship between the ejection force and the equilibrium residual die wall pressure after the fifth compression is shown in Fig. 17. Alkali metal halides containing 5% by weight of lubricant are found below the correlation line, an indication that the required forces for ejection are relatively low. Phenacetin, acetanilid, aspirin, and potassium carbonate are above the line and show that the required forces for ejection are comparatively high.

Residual elastic compressional energy remaining after the decay through a flow process in the die appears to play an important role in determining the amount of work required to remove the finished tablet.

Side wall pressure measurements seem to be considerably valuable in providing information relative to the ease of formation and characteristics of different tableting formulas.

REFERENCES

- (1) Nelson, E., *THIS JOURNAL*, **44**, 494(1955).
- (2) Windheuser, J. J., *et al.*, *ibid.*, **52**, 767(1963).
- (3) Higuchi, T., Nelson, E., and Busse, L. W., *ibid.*, **43**, 344(1954).
- (4) Higuchi, T., *et al.*, *ibid.*, **42**, 194(1953).
- (5) Nelson, E., *et al.*, *ibid.*, **43**, 596(1954).