Determination of Tablet Strength by the Diametral-Compression Test

J. T. FELL* and J. M. NEWTON†

Abstract □ The strength of lactose tablets has been measured by application of the diametral-compression test. The relative value of tensile, compressive, and shear stresses within the tablet varies, depending on the characteristics of the tablets and the surface providing the applied compression. It has been shown that to obtain reproducible results for the strength of tablets prepared at a given compression force, the tablet must break in such a manner that the tensile stress is the major stress. For a given tablet, this may require the placing of suitable padding material between the tablet and the compressing surfaces. Assessment of the type of failure can be made visually and under the correct conditions, the results expressed as a tensile strength. There are, however, a range of conditions which ensure tensile failure resulting in different values for the tensile strength. These values are characteristic of the tablet and test conditions and are not absolute values of tensile strength.

Keyphrases □ Tablet strength determination—diametral-compression test □ Tensile strength, tablets—measurements □ Compression effect—tablet fracture □ Fracture mode, tablets—testing

The authors have previously described the use of the diametral-compression test to assess the tensile strength of lactose tablets (1). This test is carried out by a procedure similar to that often used to assess the crushing strength of tablets, *i.e.*, diametral compression between two flat platens. The determination of a tensile strength, as opposed to a crushing strength, from this procedure depends upon the correct state of stress developing within a specimen of known shape and dimensions. Consider the stress distribution within a tablet, which is in the form of a cylinder, placed between the platens of a loading system. Under conditions of ideal line loading, the values of tensile σ_1, compressive σ_2, and shear stress τ can be calculated by elastic theory (2) and are illustrated in Fig. 1. The value for the maximum tensile stress σ_0 is constant over the whole of the load diameter and has a magnitude:

$$\sigma_0 = \frac{2P}{\pi D t} \qquad \text{(Eq. 1)}$$

where

P = applied load
D = tablet diameter
t = tablet thickness

The values for the compressive and shear stresses are a minimum at the center of the load diameter and infinitely high immediately under the load points (Fig. 1). These high values for shear and compressive stresses will prevent the initiation of failure in tension. In practice, ideal line loading will never occur, the load will be distributed over an actual contact area. The stress distribution across the load diameter, when the contact

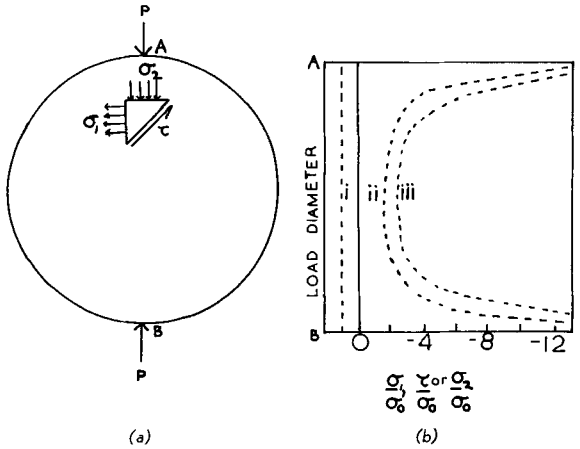

Figure 1—*Stress distribution across loaded diameter for a cylinder between two-line loads—ideal line loading (3). Key:* (a) *Loading system:* A + B = *points of loading;* σ_1 = *tensile stress;* σ_2 = *compressive stress;* τ = *shear stress.* (b) *Relative magnitude of tensile* σ_1, *compressive* σ_2, *and shear* τ *stresses compared with maximum tensile stress* σ_0. *Curve i* = σ_1/σ_0; *Curve ii* = τ/σ_0; *Curve iii* = σ_2/σ_0. P = *applied load.*

width is $^1/_{10}$ of the specimen diameter and uniform contact pressure is applied, calculated from elastic theory (2), is shown in Fig. 2. The tensile stress is constant over most of the load diameter, except for the regions near the loading area, but the shear and compressive stresses have been considerably reduced in this area. Thus, it is possible to have failure of the specimen initiated in tension, and the tensile strength can be calculated from Eq. 1. In practical terms, therefore, to obtain tensile failure of constant magnitude, the conditions of the test must ensure that a maximum length of the load diameter is under constant tensile stress, associated with minimum values for shear and compressive stresses, below the loading area.

The mechanical properties of the specimen and load platens determine the stress distribution within the specimen (3). In the case of pharmaceutical tablets compressed between metal platens, tablets are often soft compared with the platens. Hence, there will be a spreading of the load at the contact points due to flattening of the tablet, preventing line loading and reducing shear and compressive stresses. Under these conditions, failure will occur in tension and the results should be reproducible, providing that distribution of the load is not so great that the stresses within the central portion of the tablet are affected. When, however, tablets have a high elastic modulus, the conditions of ideal line loading are approached, and failure may be initiated by shear or

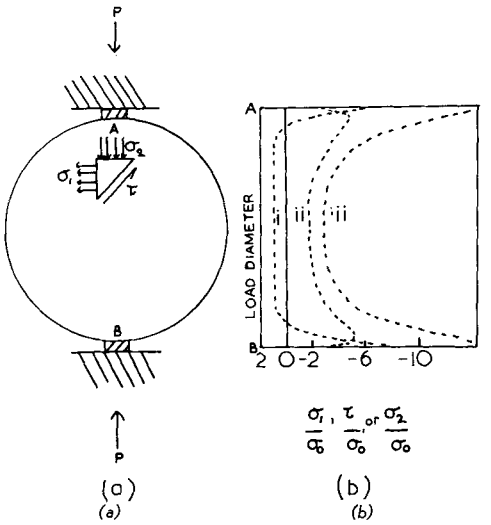

Figure 2—*Stress distribution across loaded diameter for a cylinder compressed between plates to give a constant width of $^1/_{10}$ of the diameter, with uniform contact pressure (3). Key:* (a) *Loading system:* A + B = *loading area;* σ_1 = *tensile stress;* σ_2 = *compressive stress;* τ = *shear stress.* (b) *Relative magnitude of tensile* σ_1, *compressive* σ_2, *and shear* τ *stresses compared with the maximum tensile stress* σ_0. *Curve i* = σ_1/σ_0; *Curve ii* = τ/σ_0; *Curve iii* = σ_2/σ_0. *P = applied load.*

compression. To obtain the correct conditions for tensile failure of such specimens, a narrow pad of a soft material is placed between platens and the specimen (3–6). The pad should be soft enough to allow distribution of the load over a reasonable area, minimizing shear and compressive stresses but not so soft that the distribution of load is excessive. Various suggestions have been made for the type and dimension of padding (4–6). Rudnick *et al.* (3) consider that the choice is best made from experimental observation, the type of failure being recognizable by examination of the specimen after testing. The three types of failure listed by these workers (3) are: compression and/or shear failure—here the specimen fractures in an irregular manner resulting in several irregular fragments; normal tensile failure—here the specimen splits into two halves along the loaded diameter; and triple cleft failure—the specimen splits symmetrically about the loaded diameter into four pieces. The tongue and groove shape of the outer surface and a clean central fracture are characteristics of the last method of failure. Rudnick *et al.* (3) consider this type to be a variation of the normal tensile fracture due to experimental conditions, and data from such systems can be used to calculate tensile strength. These three types of failure are illustrated by the photographs of tablets subjected to diametral compression (Fig. 3).

The applications of these principles to the determination of the strength of tablets prepared from lactose are reported in this paper.

EXPERIMENTAL

Materials—The samples of lactose used were: (*a*) a < 32-μ size fraction from a single batch of crystalline lactose (British Drug House laboratory reagent grade), (*b*) spray-dried lactose A, a < 32-μ size fraction from a single batch of spray-dried lactose (McKesson Robbins), and (*c*) spray-dried lactose, Samples B, C, and D, which were produced under known conditions using an experimental spray drier.

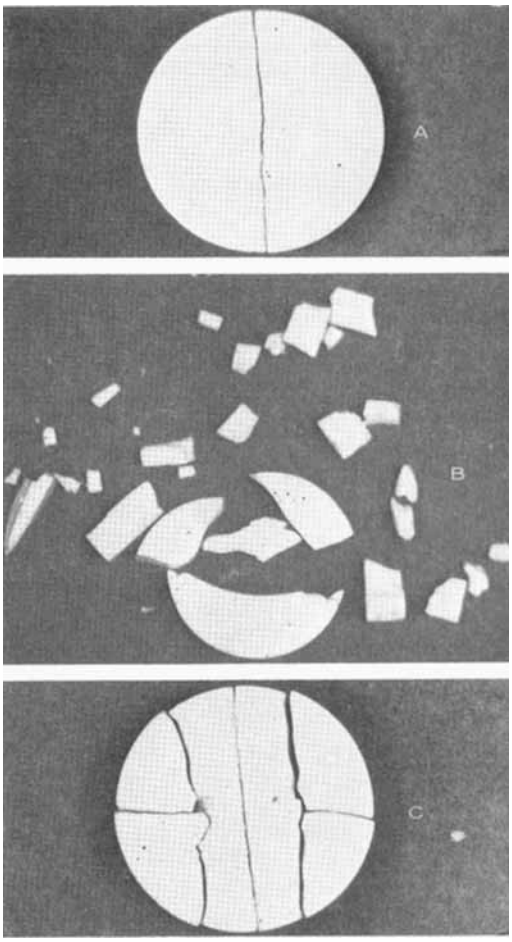

Figure 3—*Fractured tablets after diametral compression. (A) Normal tensile failure—obtained with crystalline lactose and spray-dried lactose A. (B) Shear and compressive failure—obtained with spray-dried lactose samples B, C, and D. (C) Tensile failure of spray-dried lactose samples B, C, and D using padding material.*

Methods—*Compression of Tablets*—Tablets of the different samples of lactose were prepared on an Instron physical testing instrument, floor model, modified to take a conventional tablet punch and die system [1.27-cm. (0.5-in.) diameter flat-faced punches] (Fig. 4). The powder samples (0.5 g.) were dried at 90° for 15 hr.

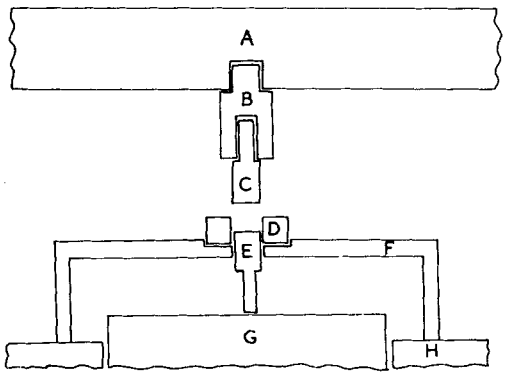

Figure 4—*Schematic illustration of modification to enable tablets to be prepared on Instron physical testing instrument. Key: A = crosshead of Instron physical testing instrument; B = upper punch holder; C = upper punch [1.27-cm. (0.5-in.) diameter flat face]; D = die; E = lower punch [1.27-cm. (0.5-in.) diameter flat face]; F = die support table; G = load cell; H = lower table of Instron physical testing instrument.*

Table I—Breaking Loads of Lactose Tablets Prepared at Different Applied Loads

Type of Lactose	Load Applied to Form Tablet, kg.	Without Padding: Mean Breaking Load of Tablet, kg.	Without Padding: Variance, V_1	Without Padding: No. of Tablets Breaking in Tension	With Padding: Mean Breaking Load of Tablet, kg.	With Padding: Variance, V_2	With Padding: No. of Tablets Breaking in Tension	Variance[a] Ratio
Crystalline	500	1.1	0.003	5	1.9	0.010	5	3.33
	1000	2.9	0.012	5	5.0	0.060	5	5.00
	2000	6.0	0.081	5	12.0	0.440	5	5.45
	3000	9.7	0.370	5	18.9	1.770	5	4.78
	4000	13.1	0.530	5	27.6	0.790	5	1.49
Spray-dried lactose A	500	2.0	0.005	5	2.7	0.011	5	2.20
	1000	4.3	0.030	5	6.7	0.050	5	1.66
	2000	8.5	0.040	5	14.8	0.970	5	24.25
	3000	13.1	0.510	5	22.3	1.900	5	3.73
	4000	17.3	0.400	5	33.1	0.640	5	1.60
Spray-dried lactose B	500	6.0	0.175	5	2.9	0.150	5	1.17
	1000	16.3	4.200	5	12.6	1.000	5	4.20
	2000	28.5	45.500	0	35.9	0.450	5	101.11
	3000	31.4	94.200	1	41.7	3.680	5	25.60
	4000	23.7	59.000	2	46.1	7.600	5	7.76
Spray-dried lactose C	500	5.3	0.150	5	6.5	0.125	5	1.20
	1000	17.0	5.700	5	22.6	1.000	5	5.70
	2000	28.8	20.050	1	41.7	1.150	5	17.43
	3000	33.7	47.100	0	53.3	4.170	5	11.29
	4000	31.1	81.600	0	62.8	3.440	5	23.72
Spray-dried lactose D	500	5.1	0.197	5	4.9	0.125	5	1.58
	1000	15.9	1.653	4	17.3	0.740	5	2.23
	2000	25.1	43.800	1	25.7	0.750	5	58.40
	3000	37.6	1.270	3	35.2	6.400	5	5.04
	4000	37.3	21.240	0	49.1	1.570	5	13.53

[a] The variance ratios are expressed as the ratio of the largest to smallest variance according to normal statistical convention ("Statistical Analysis in Chemistry and Chemical Industry," by C. A. Bennett and N. C. Franklin, John Wiley, New York, N. Y., 1954, p. 109). A significant difference, at 0.05% level, in the variance ratio is indicated by underlining the value.

and then stored in a desiccator over silica gel until required. The samples were quickly transferred to the die, previously painted with a 1% suspension of magnesium stearate in carbon tetrachloride, and compressed at a crosshead speed of 0.1 cm./min. When the required load level had been reached, the crosshead was reversed at the same speed. The tablets were carefully removed from the die, weighed, their diameter and thickness measured using a micrometer, and stored in a desiccator over silica gel until required. Tablets at a range of compression loads were produced.

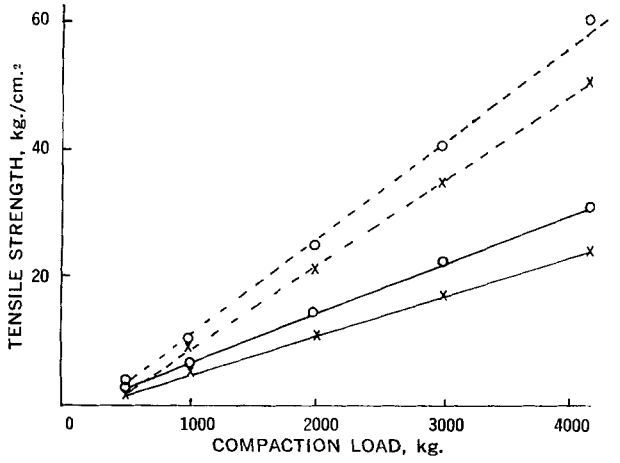

Figure 5—*The tensile strength of tablets of crystalline and spray-dried lactose A, prepared at known compression forces, when tested in the absence and presence of padding material. Key: ×, crystalline lactose; ○, spray-dried lactose A; - - -, in the presence of padding material; and —, in the absence of padding material.*

Strength of Tablets—The tensile strength of the tablets was measured by the application of the diametral-compression test described previously (1). This test consisted of compressing tablets diametrically between the platens of an Instron physical testing instrument at the rate of 0.1 cm./min. Tablets prepared from crystalline lactose and spray-dried lactose A fractured in tension between the steel platens of the Instron physical testing instrument (Fig. 3A). Tablets prepared at loads above 500 kg. from spray-dried lactose B, C, and D, however, fractured as described by Rudnick *et al.* (3) as compression or shear failure (Fig. 3B). To ensure that these tablets fractured in tension, various padding materials, *e.g.*, various thickness of paper, cardboard, and blotting paper, were placed between the tablet and the steel platens and the mode of fracture observed. Of the materials tested, it was found that three sheets of blotting paper, each 0.03 cm. thick, produced the conditions which resulted in tensile failure of the specimens. The breaking load of five tablets of each sample of lactose was determined with and without the addition of padding material.

RESULTS AND DISCUSSION

The assessment of the strength of tablets is an important aspect of the control of pharmaceutical tablets and investigations into the process of compaction. Various techniques have been used including fracture resistance (7), bending strength (8), tensile strength (9),

and crushing strength (10). Of these tests, the crushing strength is extensively used and most commercial instruments are designed to be used for this particular assessment. Brook and Marshall (11) have recently investigated the variability of such instruments, which record in different ways the load at which the tablet breaks when compressed by some means between two metal surfaces. The distribution of forces within the tablet and the related mode of fracture when tablets are tested by this procedure have not, however, been taken into account. In many cases, fracture can occur by tensile failure, resulting from correct stress conditions in the tablet. Here, variation in strength values will be due to tablet and testing instrument variation. In other cases, however, conditions of tensile failure will not apply, resulting in an additional variable of uncertain magnitude. The importance of considering the mode of fracture can be seen from the introductory remarks on the distribution of forces within the test specimen. When tablets do not break in tension in a diametral-compression test, variation in the breaking load can occur, caused by variations in the relative shear, compression, and tension forces involved. This effect is illustrated in Table I where the mean values of the breaking load of compacts when subjected to the diametral-compression test are recorded. Also given are the variance and variance ratio of the results when the tablets are tested with and without the presence of padding between the platens and the tablet. The important feature of the results is that when failure of the tablets occurs in tension, irrespective of the test conditions, there is always a low variance of the value of the load at which the tablet breaks (Table I). There is usually no significant difference, at the 5% level, between the variance of the breaking load with and without padding. When tablets do not break in tension (spray-dried lactose B, C, and D), there is a high variance of the breaking load and a significant difference in the variance of the breaking with and without padding (Table I). Only when the tablets break in tension is it possible to apply Eq. 1 and express the results for tablet strength as tensile strength.

Introduction of too soft a padding can, in fact, have the opposite effect to that just noted. Rudnick *et al.* (3) predicted from statistical theory that the amount of material subjected to a maximum tensile stress is greater when a hard rather than a soft contact surface is used. This would result in a lower strength and variance when a hard contact surface is used. This prediction was confirmed experimentally by Addinall and Hackett (12) in a detailed study of the effect of different packing pieces on the tensile strength of autoclaved plaster. The results in Fig. 5, which show the tensile strength calculated from Eq. 1, confirm these findings. The tensile strength of compacts prepared from crystalline lactose and spray-dried lactose A increased when padding was introduced. There is also a slightly greater variance of the breaking load when padding is present. In the presence or absence of padding, all tablets of these two materials fractured in tension. This raises the question as to which of the values represents the tensile strength of the tablet. Rudnick *et al.* (3) consider that there is no such value as a true tensile strength, but values obtained under any conditions are true values for those conditions. If it is required to compare the tensile strength of tablets, the conditions of the test must be the same, and tensile failure must be assured in all cases. Thus, to compare tablets prepared from spray-dried lactose B, C, and D with crystalline and spray-dried lactose A, the results in which a padding was used must be considered. For comparisons between tablets of crystalline and spray-dried lactose A, the values without padding are preferable.

A further consideration in the choice of test conditions is the actual strength of the tablets. Thus, tablets produced at low-compression forces, producing weaker tablets, often failed in tension without the need for padding. Therefore, if the tablets have a low tensile strength, it is unlikely that padding will be required.

CONCLUSIONS

The authors consider that the results indicate that it should be possible to choose test conditions, ensuring their validity by simple observation of the mode of fracture, which will improve the reproducibility of the assessment of the strength of tablets and enable meaningful comparisons to be made between the strength of tablets of different materials. For routine evaluation the results need not be expressed in terms of tensile strength, the breaking load under conditions of tensile failure being satisfactory.

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