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▲ To whom inquiries should be directed.

PHARMACEUTICAL TECHNOLOGY

Shear Cell Measurements of Powders: Determination of Yield Loci

E. N. HIESTAND▲, S. C. VALVANI, C. B. PEOT, E. P. STRZELINSKI, and J. F. GLASSCOCK, Jr.

Abstract ☐ The usefulness of the Jenike-type shear cell and associated procedure for the characterization of some powder bed properties has been established by others. An attempt to use a simpler procedure and a different shear cell and thereby to reduce the quantity of powder required is described. The yield loci obtained are very nearly linear. Multiple-regression analysis is used to obtain characteristic constants from the data for the family of yield loci. Examples of the data obtained for various pharmaceutical powders are presented.

Keyphrases ☐ Shear cell measurements—simplified determination of yield loci for powders ☐ Powder bed properties—simplified procedure and shear cell for determination of yield loci

It is postulated that the flow of powder occurs when the forces acting on a powder bed cause the resultant shear force just to exceed the powder bed's shear strength in any given direction. Therefore, the flow characteristics of the powder can be described in terms of an orthogonal set of principal forces. Only two, the major and minor forces, have a significant effect on the location of the shear plane. The magnitude of these forces depends upon the state of consolidation of the powder bed. For many applications, it is adequate to describe the changes of unconfined powder bed properties with the change in the state of consolidation. This description is in the form of a failure function, a plot of the major stress necessary to cause failure of an unconfined bed *versus* the major stress that produced the given state of consolidation.

The failure function can be estimated from shear cell data (1) or a triaxial test method (2); both methods usually fail to provide completely accurate evaluations. The shear cell methods are the most popular. This article briefly discusses the theory relating to these concepts and reports results of studies with a simple shear cell. Also included is a limited comparison of the data obtained with one sample using two different shear cells.

CONCEPTS

Figure 1 shows the direction of forces acting in both the triaxial test and the shear cell. It also shows the Mohr semicircle used to describe the stress balance at failure. With the triaxial method, the major stress, σ_m, and the minor stress, σ_s, are the applied stresses and τ and σ_n are the resultant stresses. Moreover, the angle α is observed, so the corresponding Mohr semicircle can be constructed. With the shear cell, σ_m, σ_s, and α are not determined; only τ and σ_n are known. Therefore, a single measurement does not provide enough information to permit construction of the corresponding Mohr semicircle. When σ_m and σ_s are of the correct magnitude to produce failure, *i.e.*, shearing of the powder bed, then τ and σ_n appear on the circle. As can be seen from Fig. 2, the principal stress, σ_i, which is intermediate in magnitude, does not influence the failure. The resultant τ and σ_n values represented by the Mohr semicircles involving σ_i are always less than any τ, σ_n values on the semicircle between the major and minor principal stresses. There-

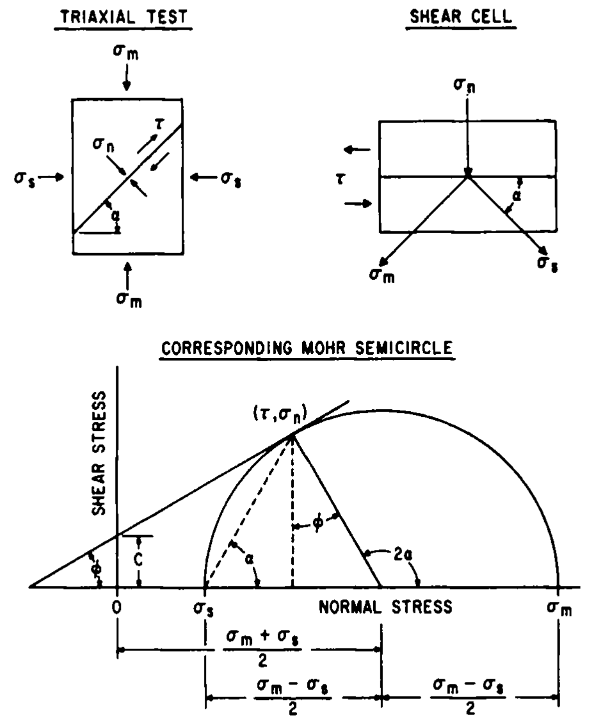

Figure 1—*Relationship of the triaxial test, the shear cell, and the Mohr semicircle. The stress equations are:*

$$\tau = {}^1/_2(\sigma_m - \sigma_s) \sin 2\alpha$$
$$\sigma_n = {}^1/_2(\sigma_m + \sigma_s) + {}^1/_2(\sigma_m - \sigma_s) \cos 2\alpha$$

Their relationship to the Mohr semicircle is obvious.

fore, the two-dimensional representation of the Mohr semicircle is adequate where the precision required is not outside the limits of the simple shear failure model.

Figure 3 is developed from the triaxial diagram, but it applies also to the shear cell. It shows the relationship between the angle α and the internal friction coefficient, which for this discussion is designated μ and is assumed to be a constant. By using any given combination of σ_m and σ_s that produces failure, the resultant magnitudes of the normal and shear stresses at any angle, α, may be calculated. These resultant stresses are σ_α and τ_α, respectively; τ_s is the shear strength of the powder bed. It is calculated from the friction coefficient, μ, the normal stress, σ_α, and an apparent cohesion, C. When τ_α is equal to τ_s, the angle of failure in shear is determined. At this point, $\mu = \tan \phi$.

A series of combinations of σ_m and σ_s can be found that produce shear failure with any given state of consolidation of a powder bed. When the corresponding τ and σ_n values are plotted, a yield locus may be drawn through the points. Each respective Mohr circle will be tangent to the yield locus, and it is this property that permits the shear cell to be used to determine α. A series of values obtained with

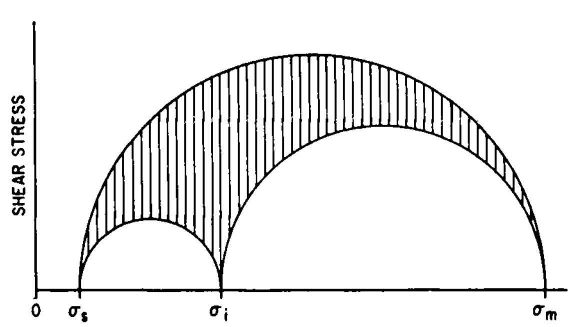

Figure 2—*The Mohr semicircles show that the largest shear stresses are produced as a resultant of the major and minor principal stresses, σ_m and σ_s, and not as a resultant of the intermediate principal stress, σ_i, with either σ_m or σ_s.*

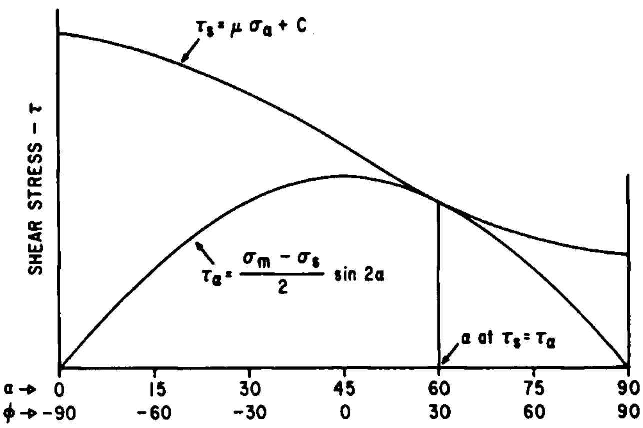

Figure 3—*Failure occurs at the angle where the resultant shear stress, τ_α, is equal to the shear strength, τ_s. Note that the shear strength results from the internal friction and cohesion. The change of both τ_s and τ_α with change in ϕ or α is shown; ϕ is the angle of friction and is equal to $2\alpha - 90°$.*

the shear cell determine a yield locus. Any corresponding combination of σ_m and σ_s can be obtained by drawing a Mohr circle tangent to the yield locus through the τ, σ_n point of interest.

SHEAR CELL DESIGN AND TREATMENT OF DATA

Probably the most popular shear cell design is that of Jenike (1). Schwedes (3) showed that the shear region in the cell has a lenticular shape. However, Williams *et al.* (2) claimed that the results are consistent with the triaxial test. Schwedes used a shear cell similar to that of Roscoe (4) and a very low, constant rate of shear. His choice of cells was, in part, based on the conclusion that the stress conditions in the Jenike cell cannot be determined.

In the authors' laboratory, the choice of cells was based on the need to work with small amounts of powders and to accumulate useful data rapidly. It was hoped that the stress distributions would be more uniform and easily defined in a thin layer of powder because the shear region would be restricted. A modification of a cell described by Nash *et al.* (5) was adopted, and a method of obtaining reproducibly a given state of consolidation was developed. The shear cell (Fig. 4) consists of a thin layer of powder between two sandpaper-covered surfaces (see *Appendix* for details). To determine a yield locus, the powder is brought to a reproducible state of consolidation by repeatedly initiating shear in the powder bed while keeping the normal load constant. The shear stress is returned to zero as soon as possible so that minimal movement occurs. After a series of shear initiations, the observed shear force becomes constant. This is called the plateau condition and is the reference state of consolidation for the powder under that specific applied load. The cell and procedures were described previously (6, 7). The plateau load is the maximum load in a given yield locus. Other points on the yield locus are obtained by reducing the load after a plateau condition has been reached; then shear is initiated just once after the load is reduced. The reduced load and resultant shear force provide one point on the yield locus, and this procedure is repeated to obtain additional points. Different yield loci are obtained by

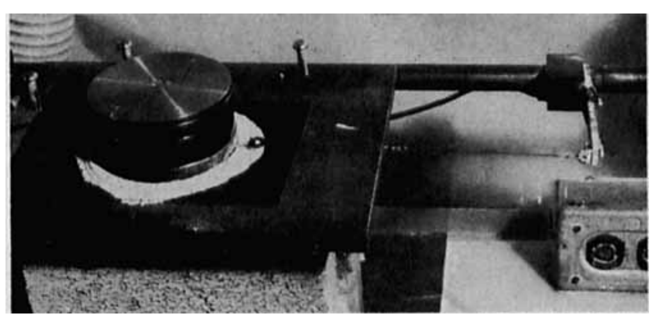

Figure 4—*Simple shear cell.*

Table I—Results of Shear Cell Studies of β-Sitosterol (40 Mesh)

	35-Point Data	Replicate 14-Point Data	14 Points from 35-Point Data
Estimate of $\tan \phi$	0.660	0.657	0.666
95% confidence interval for $\tan \phi$	0.635, 0.685	0.616, 0.698	0.626, 0.706
Estimate of $\tan \beta$	0.167	0.166	0.162
95% confidence interval for $\tan \beta$	0.146, 0.188	0.134, 0.198	0.131, 0.193
Multiple-correlation coefficient	0.997	0.997	0.992
Standard error of estimate of τ_r[a]	0.713	0.708	0.695
Estimate of b[a]	0.544	0.472	0.552
f_c *versus* σ_{pm} slope	0.245	0.243	0.237
f_c *versus* σ_{pm} intercept[a]	1.773	1.538	1.819

[a] Kilodynes/cm.^{2}.

using a different normal load to attain the plateau condition and then reducing the load from that state of consolidation.

Reducing the load after reaching a state of consolidation leads to partial elastic recovery of the powder bed. Thus, the yield locus is determined under conditions analogous to those used in evaluating other solids, *i.e.*, yield conditions under various elastic stresses; this differs from the dynamic steady-rate condition used with the other cells already discussed. This simple cell can be criticized in several ways. Obviously, it is difficult to demonstrate that the shear plane is in the powder and not at the sandpaper surface. Also, edges of the cell may not have powder under identical stress and consolidation conditions as the interior regions of the cell. However, the thin layer of powder helps to minimize these effects and, of course, requires less powder, an important consideration when working with expensive medicaments.

Powders are more complex than ordinary solids because powders have a broad range of consolidations. The shear strength changes with the state of consolidation. Thus, instead of one yield locus characterizing the powder, a family of yield loci is necessary, one for each state of consolidation. With the procedure used in this study, the plateau load is the principal factor in determining the state of consolidation. Empirically, it has been found that the family of yield loci corresponds to a simple relationship:

$$\tau_r = \sigma_r \tan \phi + \sigma_p \tan \beta + b \qquad \text{(Eq. 1)}$$

where subscript r denotes the reduced load values, subscript p denotes the plateau or consolidation condition, and $\tan \phi$ is the internal friction coefficient; $\sigma_p \tan \beta + b$ describes the apparent cohesion (C in Fig. 1), and, therefore, $\tan \beta$ is the rate of change and b is the intercept of the apparent cohesion function.

These relationships and related equations were described previously (7). Equation 1 is the classical empirical law of friction, with additional terms to account for the different states of consolidation.

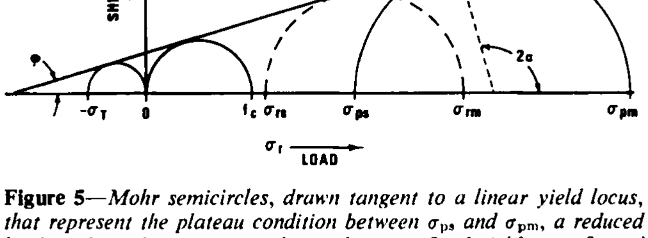

Figure 5—*Mohr semicircles, drawn tangent to a linear yield locus, that represent the plateau condition between σ_{ps} and σ_{pm}, a reduced load condition between σ_{rs} and σ_{rm}, the unconfined yield stress f_c, and the ultimate tensile strength σ_T.*

Table II—Loads Used on an 8.8-cm. (3.5-in.) Diameter Cell in Shear Cell Studies[a]

Plateau Load	Reduced Load				
3352	3109	2372	1392	902	412
(2862)	(2770)	(2170)	(1420)	(920)	(412)
(2372)	2274	1882	1392	902	(412)
(1980)	1882	(1587)	1196	804	412
(1588)	(1490)	1294	902	706	412
(1196)	1147	1000	(804)	608	412
(902)	(853)	(706)	(608)	(510)	(412)

[a] All values are in kilodynes. The 14-point data are indicated by parentheses.

The individual yield locus is associated with only one state of consolidation. Therefore, it is not surprising that it is linear[1] since the conditions become identical to those between two large pieces of solid, where the linear relationship is the accepted and expected result. Different equations have been used with data obtained with the Jenike shear cell. The Warren Springs equation[2] is said to provide adequate flexibility in describing nonlinear yield loci such as those obtained in the dynamic conditions used with the Jenike cell (8). However, it would have to be modified to incorporate the large tensile strength values observed here. The simplicity of a linear relationship permits other interesting relationships (*e.g.*, Eqs. 2 and 3) to be derived readily (7).

In the authors' laboratory, values of τ_r, σ_r, and σ_p are used in a multiple-regression analysis[3] to obtain estimates of $\tan \phi$, $\tan \beta$, and b. These values may be used to determine the failure function and the ultimate tensile strength function. Equation 2 is the former and Eq. 3 is the latter:

$$f_c = \left[\frac{2 \cos \phi \tan \beta}{1 + \cos \phi \tan \beta}\right]\sigma_{pm} + \frac{2b(1 + \sin \phi)}{\cos \phi\,(1 + \cos \phi \tan \beta)} \qquad \text{(Eq. 2)}$$

$$\sigma_T = \left[\frac{1 - \sin \phi}{1 + \sin \phi}\right] f_c = \left[\frac{2(1 - \sin \phi)^2 \tan \beta}{\cos \phi(1 + \cos \phi \tan \beta)}\right]\sigma_{pm} + \frac{2b(1 - \sin \phi)}{\cos \phi(1 + \cos \phi \tan \beta)} \qquad \text{(Eq. 3)}$$

Equations 2 and 3 are derived from Eq. 1 and the geometric relationships shown in Fig. 5; σ_{pm} is the major consolidation stress, f_c is the unconfined yield stress (the major principal stress when the minor one is zero); and σ_T is the ultimate tensile strength. The ultimate tensile strength is the strength a powder bed would have in tensile failure if conditions comparable to those in the shear cell could be

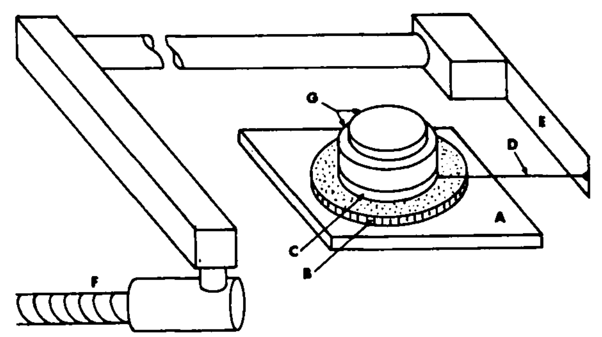

Figure 6—*Arrangement of apparatus. Key: A, bottom plate of shear cell; B, powder bed; C, top plate of shear cell; D, tow line; E, strain gauges; F, jack (motor driven); and G, weights.*

[1] Some deviation from linearity is observed. However, for many materials (*e.g.*, β-sitosterol) the tensile strength is too large to conform to the curvilinear model. Therefore, the linear model has been proposed.

[2] The Warren Springs equation is of the form $(\tau_r/C)^n = (\sigma_r/S) + 1$ where τ_r, σ_r, and C have the same meaning as already given, S is the tensile strength, and n is an empirical constant.

[3] The point (τ_p, σ_p) usually is below the corresponding yield locus. Therefore, it is not used in the multiple-regression analysis. A procedure analogous to that used by Jenike in a similar situation (1) is used here; *i.e.*, a (τ_r, σ_r) point is determined where σ_r is approximately 95% of σ_p. This is the largest value used in evaluating an *individual* yield locus.

Table III—Tan ϕ Calculated by Least Squares for Each Individual Yield Locus[a]

Plateau load	3352	2862	2372	1980	1588	1196	902
tan ϕ	0.665	0.653	0.654	0.642	0.660	0.582	0.575

[a] Based on 35 load conditions shown in Table II.

Table IV—Comparison of Two Different Lots of the Same Chemical Produced during the "Scale-Up" of a New Drug

	Ambient Conditions (as Received)		Equilibrated with a 50% Relative Humidity Environment	
Function	Lot A	Lot B	Lot A	Lot B
tan ϕ	0.761	0.688	0.714	0.689
tan β	0.206	0.155	0.238	0.120
b[a]	1.90	0.795	1.562	0.792
f_c *versus* σ_{pm} slope	0.283	0.175	0.324	0.180
f_c *versus* σ_{pm} intercept[a]	6.602	2.721	5.088	2.738

[a] Kilodynes/cm.^{2}.

realized in a tensile strength measurement. Of course, the value also is based on the assumption that the yield locus is linear into the tension region so that the extrapolation is valid; this is not easily demonstrated. However, comparison of the tensile strengths calculated from shear cell measurements with values obtained using the diametral compression method of evaluating compacts provides strong evidence that, at least in some cases, the extrapolation is not grossly in error (7).

RESULTS AND DISCUSSION

To illustrate the reproducibility of evaluations of powders with this cell, some experimental results are included in the tables. Table I shows the observed properties of a β-sitosterol sample, and Table II shows the normal load forces used in this study. Column 1 of Table I shows the results of using a 35-point determination. The middle column is for a duplicate determination in which only 14 points were collected. The normal loads for these 14 points are shown in parentheses in Table II. The last column of Table I shows the results when the corresponding 14 data points (those in parentheses) are selected from the 35-point data. The 35 points defined seven yield loci. Table III shows the values of these slopes, tan ϕ, for each individual yield locus. As illustrated by this example, tan ϕ often is less for small conditioning loads. Note that the multiple-regression analysis yields a value for tan ϕ characteristic of the yield loci for larger conditioning loads. The slope and intercept values for the corresponding failure functions, Eq. 2, are given at the bottom of Table I. These variations are larger than desired but they are believed to be respectable when one allows for the great difficulties experienced in evaluating powders.

Table IV illustrates the usefulness of the shear cell data. Two different, small production lots of a new drug were compared. Shear cell measurements were not necessary to reveal that the flow properties of the two lots were different. Lot A would not fall through a coarse screen without agitation, but Lot B fell through unless carefully placed on the screen. Obviously, both lots had poor—but not equally poor—flow properties. Both, the slope and intercept of the failure function, f_c *versus* σ_{pm}, indicate poorer flow characteristics for A than for B. Thus, the shear cell study measured quantitatively the differences. It also established that these differences were not due just to the equilibration with different atmospheres. Obviously, it is important to determine what causes variations such as those in Table IV, but the ability to measure meaningful flow properties must come first. Unfortunately, small differences in measured values are not always significant. The determinations are not accurate to the three significant figures shown in the tables, but the multiple-regression analysis yields the associated confidence limits to serve as guides to the significance of the values.

Table V shows the values obtained in two different equilibrium humidity environments for sucrose, starch, and a mixture of the two powders. It is obvious that the starch is a less cohesive powder than the sucrose, especially at the lower humidity. Furthermore, the starch is effective at the 25% level in imparting low cohesiveness to the mixture when the humidity is low. At higher humidity, the influence is less. Again, these data are included to illustrate the shear cell method of evaluation. No explanation of the observed effects are established at this time.

The only data available to the authors are inadequate for a thorough comparison of the results with the simple shear cell method described here and the Jenike method. However, the available data are included to provide a limited comparison and to illustrate some problems in evaluating powders. A powder mixture was submitted to a commercial laboratory, one that uses the Jenike cell and procedures. They made only a six-point determination, three points on each of two yield loci, and reported the failure function graphically. It was equivalent to $f_c = 0.186\ \sigma_{pm}$. The values of f_c and σ_{pm} were obtained by drawing Mohr semicircles tangent to the yield loci. For comparison, the multiple-regression analysis method was applied to their data (Table VI). In the authors' laboratory, the simple shear cell was used to obtain a 40-point determination on the same lot of material (Table VI). It is difficult to compare 40-point and 6-point data except through statistical values, such as in Table VI. It is obvious that improved precision would be desirable. The simple cell used in this study was only 5.1 cm. (2 in.) in diameter. Later, the diameter was increased to 8.8 cm. (3.5 in.) and significant improvement in precision resulted. The other data reported in this paper were obtained with the larger cell.

The graphs submitted by the commercial laboratory showed that one data point had been omitted in their analysis. The last column of Table VI shows the results of the multiple-regression analysis when the same point is omitted. Since only three points were available to determine a yield locus, rejection of one left only two. The criterion for such a selection is not obvious, but certainly it improves the multiple-correlation coefficient. Also, the calculated failure function corresponds much more closely to the one reported graphically.

CONCLUSION

The examples discussed demonstrate that the simple shear cell can be used to provide characterization of the unconfined yield strengths of powders. It is not anticipated that the results will be identical to those obtained by different procedures. However, the simplicity of the system and the use of small quantities of powder make this approach attractive. In time, definitive work may determine which procedure and cell yield the most accurate data.

Table V—Effect of Mixing Two Powders

	Sucrose	Starch[a]	75% Sucrose–25% Starch
	Relative Humidity 25%		
tan ϕ	0.672	0.704	0.665
tan β	0.162	0.054	0.132
b[b]	1.058	9×10^{-3}	5.6×10^{-2}
f_c *versus* σ_{pm} slope	0.237	0.0838	0.199
f_c *versus* σ_{pm} intercept[b]	3.49	3.2×10^{-2}	0.187
	Relative Humidity 50%		
tan ϕ	0.663	0.655	0.650
tan β	0.216	0.081	0.167
b[b]	1.024	0.187	0.794
f_c *versus* σ_{pm} slope	0.305	0.127	0.246
f_c *versus* σ_{pm} intercept[b]	3.237	0.645	2.560

[a] StaRx 1500. [b] Kilodynes/cm.^{2}.

Table VI—Comparison of Jenike Cell Data with Data from a Simple Shear Cell

	40 Points, Simple Cell	6 Points, Jenike Cell	5 Points, Jenike Cell
Estimate of tan φ	0.653	0.784	0.720
95% confidence interval for tan φ	0.640, 0.667	0.492, 1.076	0.520, 0.868
Estimate of tan β	0.068	0.054	0.111
95% confidence interval for tan β	0.056, 0.080	−0.105, 0.213	0.021, 0.201
Estimate of b^a	5×10^{-3}	23.2	4.46
Multiple-correlation coefficient	0.999	0.989	0.999
Standard error of estimate of $\tau_r{}^a$	1.11	18.0	6.21
f_c *versus* σ_{pm} slope	0.108	0.082	0.165
f_c *versus* σ_{pm} intercept^a	2×10^{-2}	91.5	16.0

^{a} Kilodynes/cm.^{2}.

APPENDIX: SHEAR CELL AND POWDER BED PREPARATION

A 12- by 16-cm. piece of 50-grit X-weight aluminum oxide emery cloth is bonded (using double-sided pressure sensitive tape) to a level, heavy piece of steel plate to form the bottom element of the shear cell. A 12.4-cm. diameter disk of the same material is bonded to the bottom of a 12.4-cm. diameter brass disk to form the upper element. A small steel hook is silver soldered to the bottom of the edge of the brass disk to provide a connection for pulling as near to the shear plane as possible.

A hole of 10.5-cm. diameter is made in a piece of aluminum plate to serve as a template for forming the powder bed. Plates of various thicknesses are available, permitting a choice in powder bed thickness. The initial thickness of the powder bed usually is 0.31 cm. (0.125 in.) with coarse materials and 0.15 cm. (0.06 in.) with fine materials. The template is placed over the bottom emery cloth, and powder is sifted into the hole of the template. A spatula is used to scrape across the plate to level the powder while avoiding packing of the bed. Excess powder is pushed out of the way and the template is removed. The top element of the shear cell is placed gently onto the powder bed, and the steel tow line from the strain gauge is placed over the hook. Care is exercised to assure that the pulling motion is parallel to the plane of the powder bed (Fig. 6).

A series of thin brass weights are made from brass cylinders to facilitate stacking and removal of selected weights. After placing the desired weights on the top element of the cell, shear is induced by moving a cantilever-type strain gauge. The elastic deflection of the gauge beam is 6.2 mm./kg. The magnitude of this deflection influences the movement of the top element of the shear cell each time shear is initiated. Excess movement affects the magnitude of stress on the next shear initiation.

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▲ To whom inquiries should be directed.