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ADVANCED RESEARCH IN SOLIDS TRANSPORT:
RHEOLOGICAL BEHAVIOR OF DENSE SUSPENSIONS

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1. SUMMARY

The objective of this program is to develop a basic understanding of the fundamental rheological properties of high solids concentration (50% to 100%) slurries and wet cake.

During this quarter the goals of Task 3 were advanced. The dynamic shear cell was completed and shakedown experiments begun. In order to have a basis for comparison with published technology experiments were performed with the Jenike shear cell and glass beads. Experiments were also performed with dry glass beads in the dynamic shear test cell.

The Conclusions, plans for the next quarter, and the experiments are described in the following sections.

1.1 CONCLUSIONS

1. The dynamic shear cell is complete and operational.
2. Jenike shear cell experiments have been performed and the results compare quite well with data reported in the literature.
3. Several experiments have been performed to obtain operating information and develop ideas for establishing an operating procedure for the dynamic shear cell.
4. Large shear stresses must be overcome in order to shear the packed glassbeads in the dynamic shear cell.
5. Shear stress is independent of speed and direction.
6. Shear stress is linearly proportional to the normal stress, however, it is not certain that this is the true yield stress.
7. A distinct shearing zone is observed.

8. When the yield stress obtained with the dynamic shear tester is plotted with similar data from the Jenike shear cell these compare well with data reported in the literature.

1.2 PLANS FOR NEXT QUARTER

1. Develop a procedure for loading and consolidating material in the dynamic shear cell.
2. Perform shear stress calibrations with the dynamic shear cell loaded with glass beads and water.
3. Expand the experiments to include smaller glass beads and mixtures of the large and small beads with water.

2. TASK 3 - DESIGN CONSTRUCTION AND SHAKEDOWN OF THE EXPERIMENTAL APPARATUS

2.1 THE DYNAMIC SHEAR CELL

A cross section of the dynamic shear cell is shown in Figure 2.1. The lower portion of the shear cell rotates while the top section is stationary. Test material is loaded in the annular section. A compressive load is applied with a weighted disc placed on top of the test material. The distance between the vanes in the upper and lower annulus section define the shear zone "h".

A picture of the dynamic shear cell is shown in Figure 2.2. The test cell is mounted on a steel base. A variable speed motor and gear reducer drives the test cell at rates up to 300 rpm. The AC inverter mounted on the wall functions as a programmable controller that provides soft starts, predetermined ramp times and controls speed. Load cells mounted on the circumference of the stationary section provide torque data. A laser system, adjacent to the test cell, can be used to study near wall particle motion through a quartz window in the stationary section. Screw jacks mounted on the plate supporting the test cell are used to adjust the position of the test cell relative to the laser. Data is collected with a personal computer and displayed on a x-y recorder.

2.2 THE DYNAMIC SHEAR CELL EXPERIMENTS

Shear cells have been used for measuring the shear stress of solid-liquid mixtures. Even though it does not satisfy the requirements of a rheometer, shear cells have been accepted as reasonable devices. The error due to centrifugal effects is much smaller than experimental error, thus it is usually neglected [4].

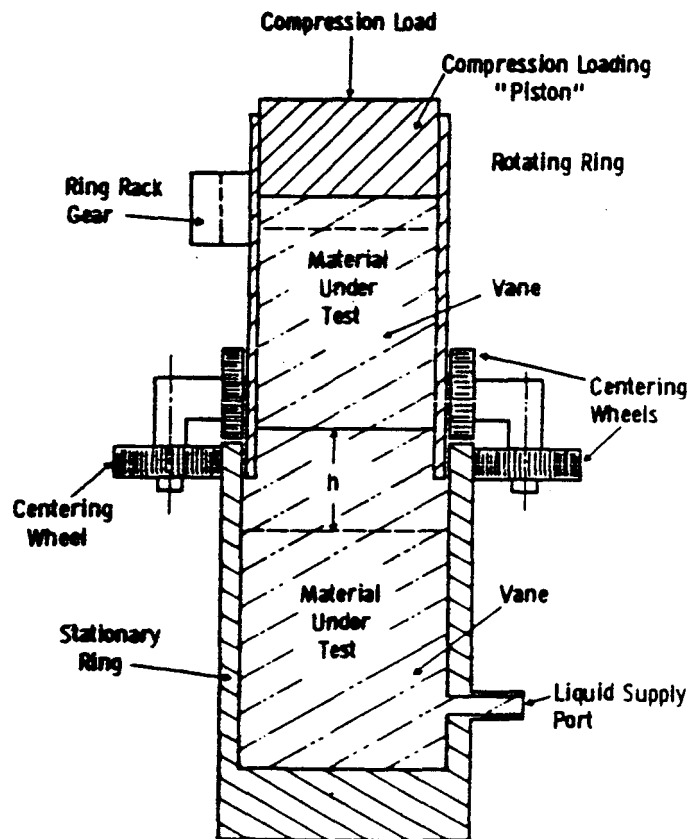


Figure 2.1 — Cross Section of the Dynamic Shear Cell

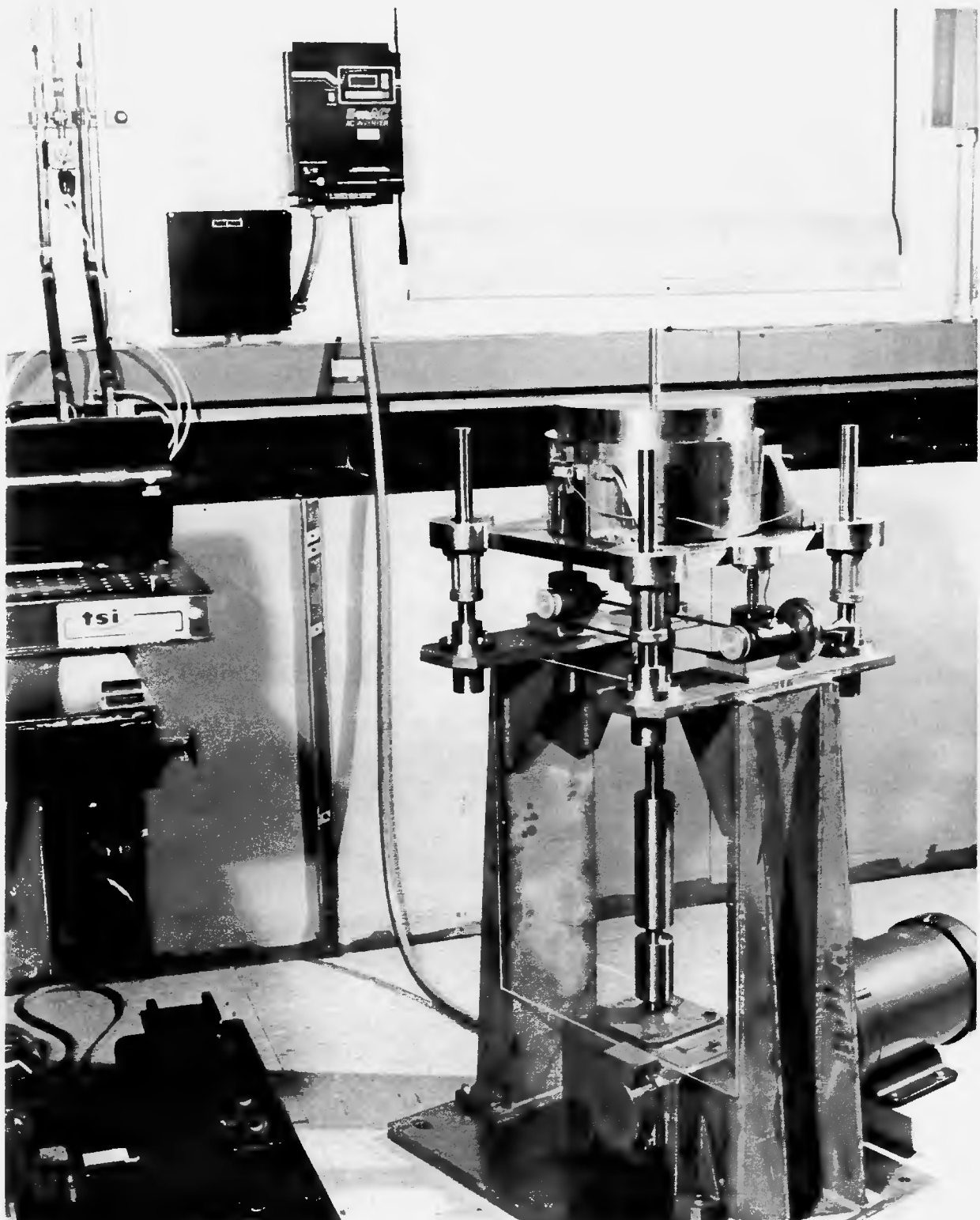


Figure 2.2 — The Dynamic Shear Cell

Our dynamic shear cell is different from others. Thus it is necessary to establish a procedure for operating this apparatus. The purpose of these experiments is to obtain information on the characteristics of the shear cell, to calibrate the apparatus, and then to establish the operating procedure. To accomplish these test goals each experiment is divided into three steps: no load test in one direction, no load test in the opposite direction, and a shear test.

The experimental procedure is described in detail in Appendix A.

2.2.1 No Load Test I

The torque measured in this test is shown in Figure 2.4 and summarized in Table 2.1. Although the mean torque value is nearly constant, large fluctuations are observed at low speed. This is due to the variation in motor speed which is a dynamic characteristic of the AC motor. As the motor speed increases, the high torque fluctuations vanish; instead a coherent variation is observed, whose pattern is nearly the same regardless of the speed. This is caused by the friction between the upper and lower cell.

Residual torque is measured by the torque load cell. When the lower cell stops rotating, residual force remains in the load cell due to static friction. Thus, it is not certain at this point if the torque shown in Figure 2.3 is the true torque, free from residual torque.

2.2.2 No Load Test II

Even though the load cell and recorder are calibrated accurately, the residual torque causes inaccurate torque readings when the test is repeated or the direction of rotation is reversed. Thus it is necessary to evaluate accurately the residual and the no load torque.

If we model the load cell and shear cell as a spring-mass system, the torque in the load cell varies as shown in Figure 2.4. In that figure, T_f and T_r are the friction torques in the forward and reverse directions, respectively, and T_1 and T_2 are the residual torques

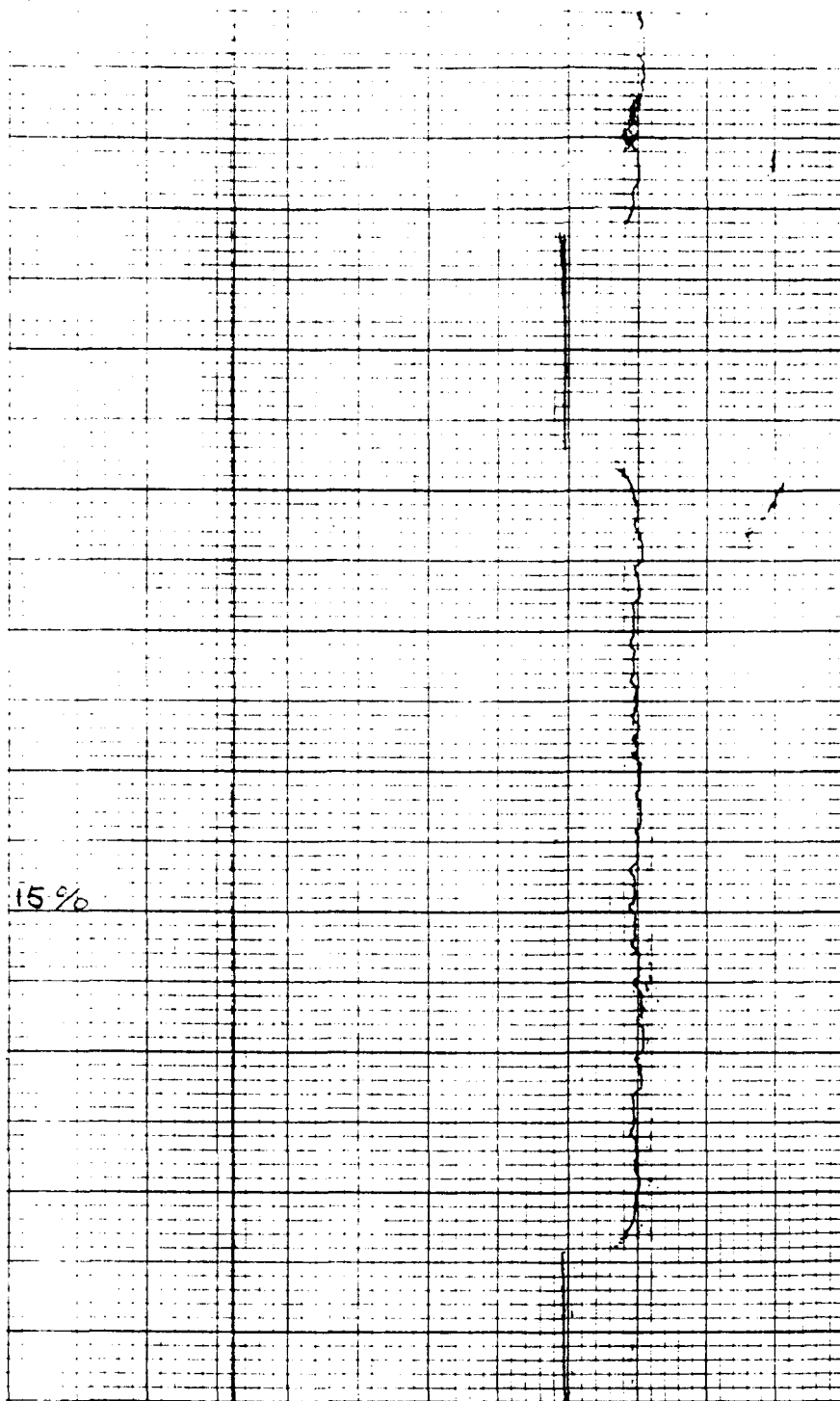


Figure 2.3 — Sample Output of Idle Test I.

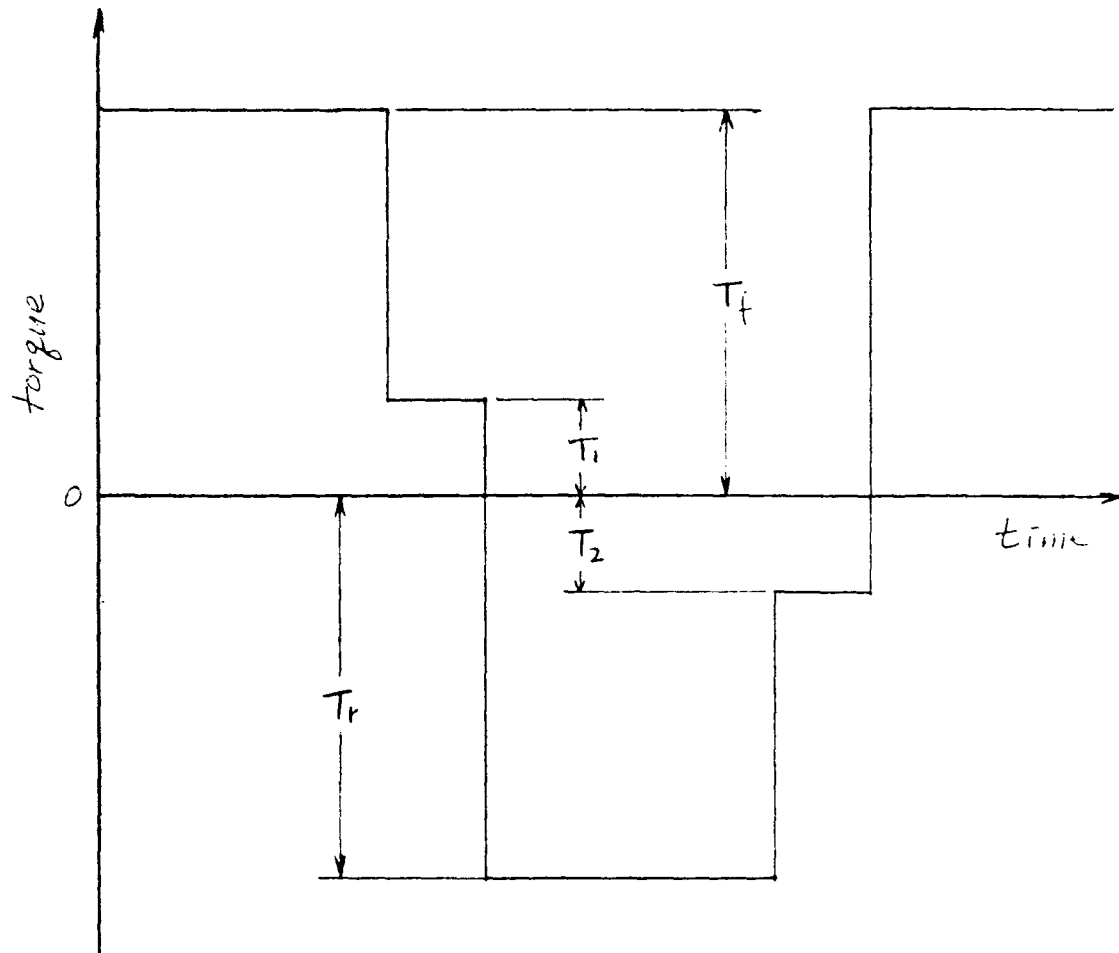


Figure 2.4 — Idealized Torque Trajectory.

Table 2.1 — No Load Torque Test

Speed (%)	Mean Torque (in-lbs)	Dev. of Torque (±) (in-lbs)	Dev. %
1	31	7	23
2	36	5	14
3	50	5	10
4	53	5	9
5	50	5	10
6	52	5	10
7	48	4	8
8	53	5	9
9	50	5	10
10	53	4	8
15	53	4	8
20	53	4	8
25	53	4	8
30	53	4	8

in the forward and reverse direction, respectively. Actual torque is obtained by dividing the distance between T_f and T_r by 2. Residual torque is calculated in the same manner.

In this test, the direction of cell rotation is changed several times, while keeping the speed constant at 20% (~ 59.3 rpm). All other parameters are the same as used previously.

Results

The variation of torque is shown in Figure 2.5. From that, we obtain the following:

mean torque = 70 in-lb.,
deviation in torque = ± 5 in-lb.,
residual torque = 15 in-lb.,

The zero-torque point can be found as a midpoint between the forward torque and reverse torque.

2.2.3 Shear Test

The purpose of this test is to investigate the performance of the shear cell with glass beads, and to observe the behavior of the material in the gap containing the shear zone.

Test Conditions

material: glass bead (mean diam. = $80 \mu\text{m}$)
gap height: 1 cm
consolidation: Not applied.
motor speed and load: see Table 2.2.

Results

The torque records are shown in Figure 2.6, and results of the shear test are summarized in Table 2.3.

From the experiment the following observations can be made:

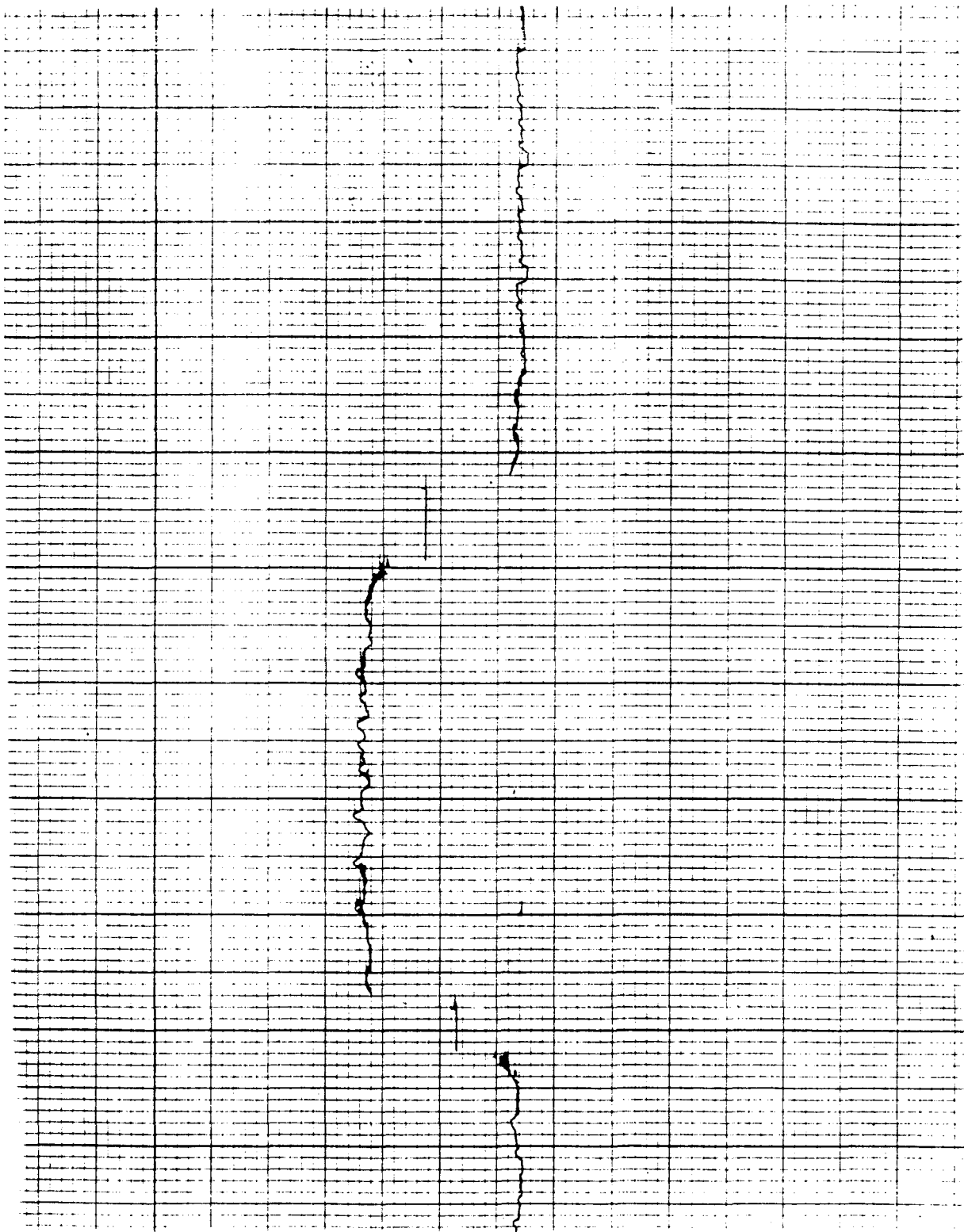


Figure 2.5 — Sample Output of No Load Test II.

Table 2.2 — Speed and Load of Shear Test		
Test #	Cell Speed (RPM)	Load (lb)
#101	30	21
#102		32
#103		54
#201	15	10
#202		32
#203		54
#301	15	54
#302		32
#303		10
#401	60	10
#402		32
#403		54

Table 2.3 — Result of Shear Test		
Test #	Mean Torque (in-lb)	Deviation (\pm in-lb)
#101	40	+45., -3.
#102	65	
#103	75	
#201	25	1.
#202	44	2.
#203	75	3.
#301	73	3.
#302	42	2.
#303	25	1.
#401	26	1.
#402	42	2.
#403	67	2.

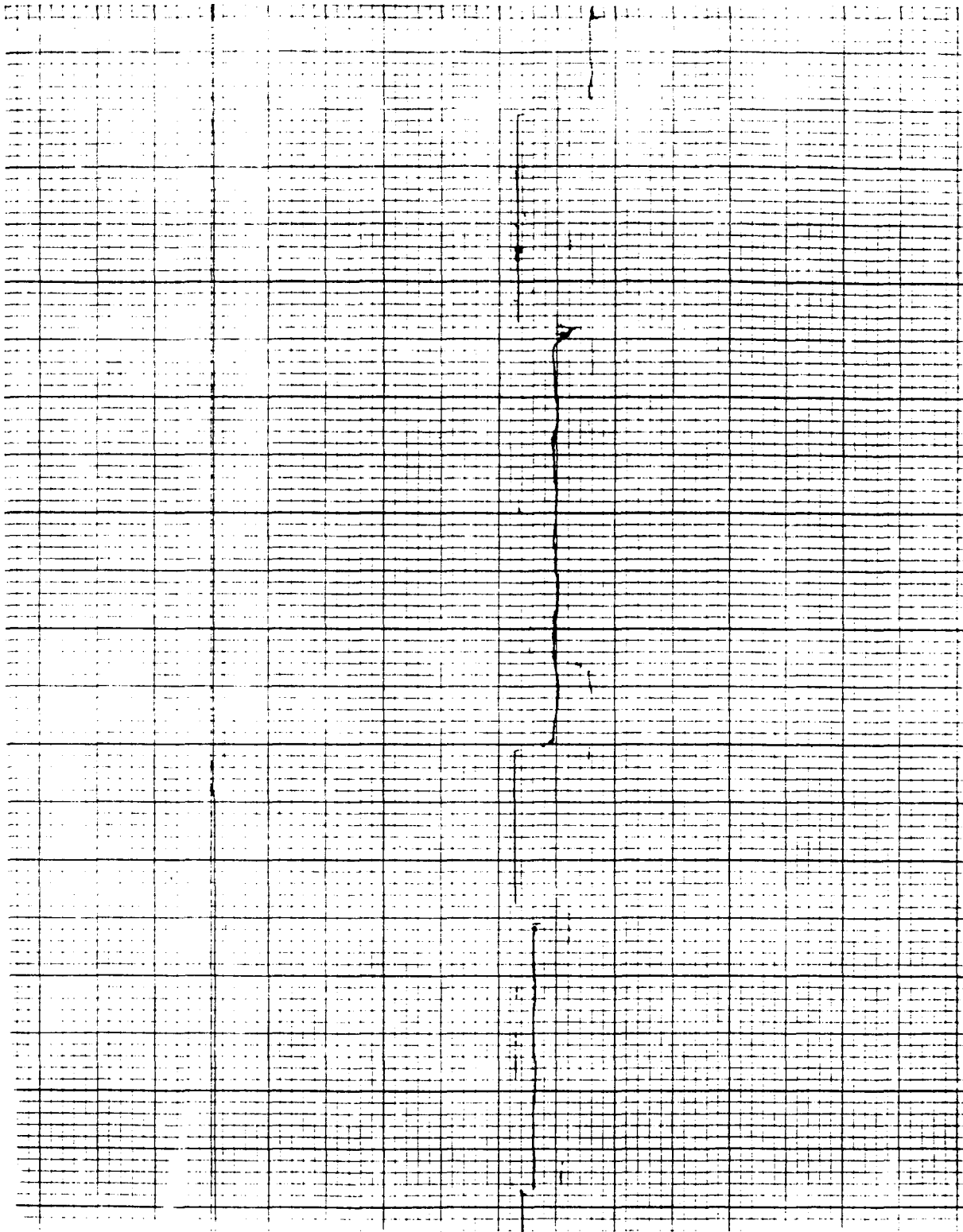


Figure 2.6 — Sample Output of Shear Test (#2xx).

1. Before uniform shear is achieved, a large torque is needed to overcome the initial deformation.
2. After uniform shear is obtained the torque is nearly uniform but decreasing slightly.
3. Torque is independent of speed and direction.
4. As the test proceeds, torque fluctuation decreases.

The thickness of the shearing zone was less than the shear gap height. Several torque values are less than the no load torque, which means that the glass beads penetrate into the gap between the lower and upper cells and then act as ball bearings. Consequently, it is necessary to know the torque due to the shear cell itself when the glass beads are loaded. One test was performed in which the torque was measured after the glass beads were loaded slightly above the lower cell and the rotation direction was reversed several times. Unfortunately, the expected information was not obtained since the material did not penetrate into the gap.

Even though the torques recorded include the friction of the shear cell, they are sufficient to give the shape of the yield locus. In order to get the yield locus the data must be examined as follows. First shear stress is converted into pascal.

Area of shear plane (A_s) = 236 cm²

Effective radius (R_e)

$$Re\pi (R_2^2 - R_1^2) = \int_{R_1}^{R_2} 2\pi r^2 dr$$

$$\begin{aligned} Re &= \frac{2(R_2^3 - R_1^3)}{3(R_2^2 - R_1^2)} & (2-1) \\ &= \frac{2(14^3 - 11^3)}{3(14^2 - 11^2)} \\ &= 12.56 \text{ cm} \end{aligned}$$

Then, shear stress: ReAs $\tau = T$

$$\begin{aligned}\tau &= \frac{T}{ReAs} \\ &= \frac{(2.54)(4.448)(10^4)}{(12.56)(236)} \\ &= 38.12 \text{ T Pa}\end{aligned}$$

If we select one test series (#3xx) as the representative, then the information in Table 2.4 is the result.

Table 2.4 — Representative Shear Stress

Normal Load (lb)	Normal Stress (Pa) (N/M ²)	Shear Stress (Pa) (N/M ²)
54.	10178.	2783.
32.	6031.	1601.
10.	1885.	953.

We have not confirmed that these shear stresses are true yield stresses. However, it is worthwhile to plot them with the results of the Jenike test as a comparison. This is seen in Figure 2.7. The magnitudes of the shear stresses are less than the shear stress obtained with the Jenike test. A similar trend is reported in [5].

2.3 THE JENIKE SHEAR CELL EXPERIMENTS

The objective of this work is to develop baseline information on glass beads with well developed devices in order to compare with data obtained with the new dynamic shear experiment. The Jenike Shear cell is used to develop basic information on dry glass beads.

The test procedure used is from Reference 1 and is described in Appendix B.

COMPARISON OF JENIKE AND SHEAR CELL

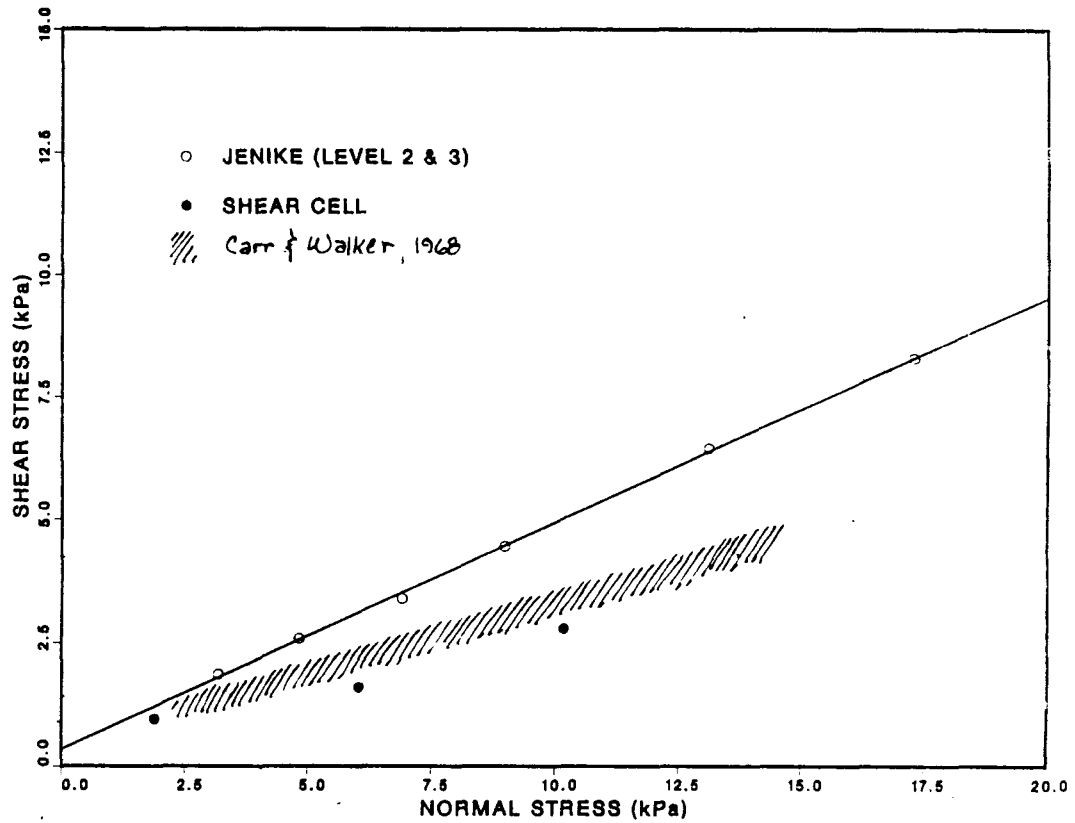


Figure 2.7 — Comparison of Jenike and Shear Cell Tests.

Each test was repeated several times. For preconsolidation, the twisting angle was 30°. Thirty twists were optimum for levels 1 and 2 (W = 27.5 Kg and 18.5 Kg respectively) and 50 twists optimum for level 3 (W = 7 Kg).

A typical chart recording is shown in Figure 2.8. and data obtained from tests are shown in Table 2.5 Data manipulation will be explained in the following section.

2.3.1 Data Reduction

Weights designated in the table above are not true normal forces, but the weight of ring, its enclosed material, and the cover must be added.

$$V = W + W_o$$

$$\text{or } \bar{V}_i = \bar{W}_i + W_o$$

In this experiment, $W_o = 0.5 \text{ kg}$.

V = True normal load (Kg)

W = Consolidation load (Kg)

W_o = Weight of ring, cover and solid above the shear plane

\bar{V} = True consolidation load

$\bar{}$ = Value during shear testing.

In each test, if over- or under-consolidation is observed the corresponding shear force is discarded. Then remaining shear forces are averaged. These data are shown in Table 2.6.

The standard deviations (σ) for the steady state shear forces during consolidation (S) are 3%-4% which is considered acceptable. Since the steady state values are not the same but fluctuate slightly, prorating is performed. The shear forces are prorated using the mean of the steady state values (S) as the reference value.

S = Test shear force (Kg)

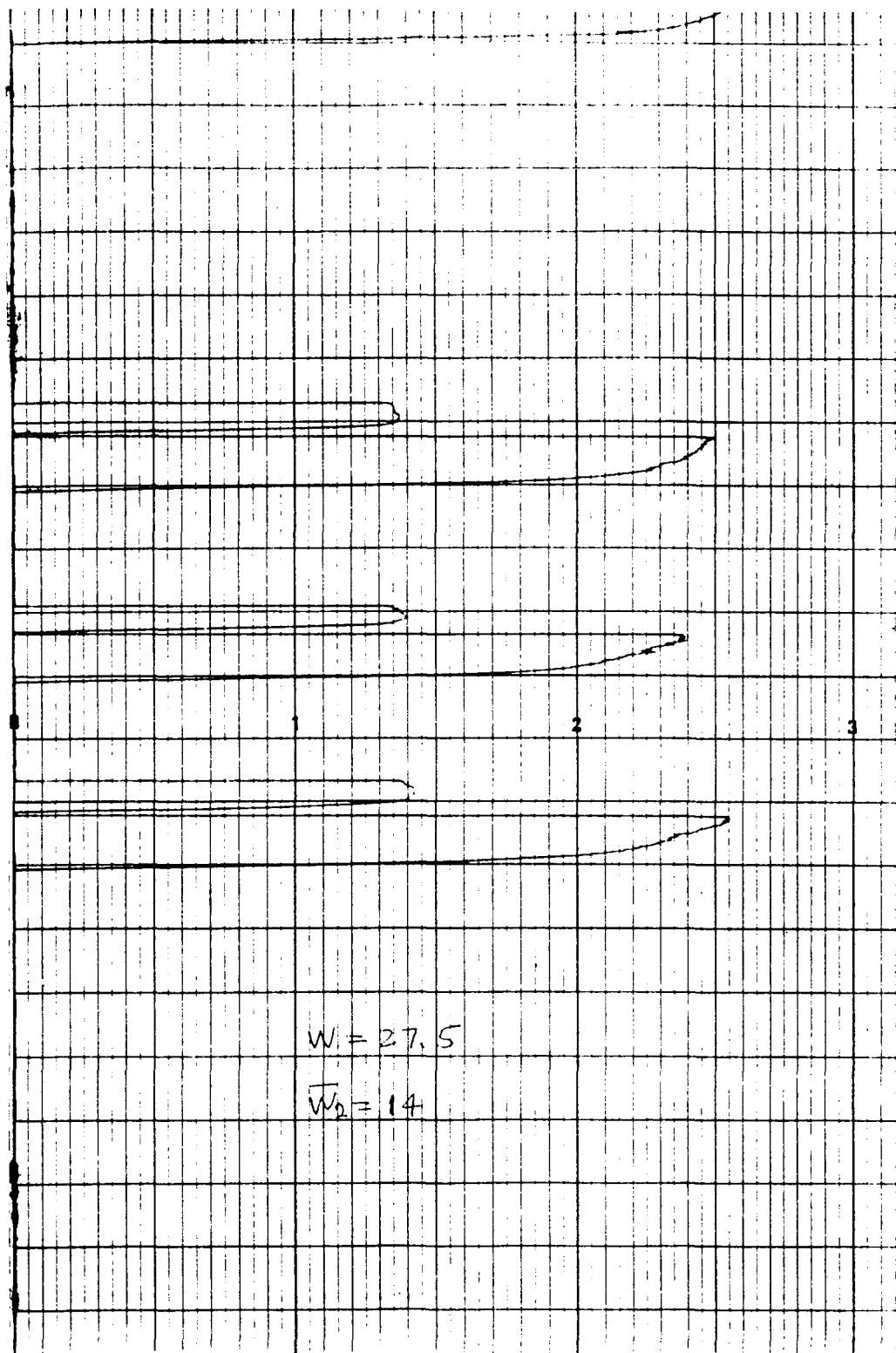


Figure 2.8 — Sample Output of Jenike Test.

Table 2.5 — Data from Instantaneous Yield Locus Test

W (Kg)	S (Kg)	\bar{W}_i (Kg)	\bar{S}_i (Kg)
27.5	12.20	18.5	8.65
	13.35		9.20
	13.00		9.05
	Under		8.95
	12.75	14.	7.10
	11.90		7.00
	Under		6.85
18.5	13.15	9.	4.75
	12.15		4.55
	12.90		4.80
	Under		6.10
	8.75	12.	6.10
	8.60		6.00
	8.75	9.	4.63
	8.10		4.50
	Under		4.60
7.	8.40	6.	3.15
	Under		3.35
	8.90		3.35
	3.23	4.5	2.46
	Over		2.50
	Under		2.52
	3.17		2.53
		3.	
	3.07		1.83
	Under		1.83
	3.18		1.90
	3.00	1.8	1.30
	Over		1.32
	3.13		1.34

Table 2.6 — Averaged Data

V (Kg)	S (Kg)	\bar{V}_i (Kg)	\bar{S}_i (Kg)	\bar{S} (Kg)	σ (Kg)
28	12.85	19.0	8.97	12.68	0.49
	12.33	14.5	7.05		
	12.73	9.5	4.70		
19	8.68	12.5	6.05	8.58	0.27
	8.43	9.5	4.57		
	8.65	6.5	3.25		
7.5	3.20	5.0	2.50	3.13	0.08
	3.13	3.5	1.87		
	3.07	2.3	1.32		

Level 1

$$\bar{S}_1 = 8.97 \times 12.68/12.85 = 8.85$$

$$\bar{S}_2 = 7.05 \times 12.68/12.33 = 7.25$$

$$\bar{S}_3 = 4.70 \times 12.68/12.73 = 4.68$$

Level 2

$$\bar{S}_1 = 6.05 \times 8.58/8.68 = 5.98$$

$$\bar{S}_2 = 4.57 \times 8.58/8.43 = 4.65$$

$$\bar{S}_3 = 3.25 \times 8.58/8.65 = 3.22$$

Level 3

$$\bar{S}_1 = 2.50 \times 3.13/3.20 = 2.45$$

$$\bar{S}_2 = 1.87 \times 3.13/3.13 = 1.87$$

$$\bar{S}_3 = 1.32 \times 3.13/3.07 = 1.35$$

Values of yield point and steady state limit are summarized in Table 2.7.

Table 2.7 — Yield Points

	Level 1	Level 2	Level 3
(V,S)	(28.0,12.68)	(19.0, 8.58)	(7.5,3.13)
(\bar{V}_1, \bar{S}_1)	(19.0,8.85)	(12.5,5.98)	(5.0,2.45)
(\bar{V}_2, \bar{S}_2)	(14.5,7.25)	(9.5,4.65)	(3.5,1.87)
(\bar{V}_3, \bar{S}_3)	(9.5,4.68)	(6.5,3.22)	(2.3,1.35)

Yield points obtained are plotted in Figures 2.9 through 2.12 by means of "DISSPLA", a package for plotting. The yield locus for each consolidation level is obtained by using the least square method. Check

of validity follows Figure 1 of reference 1. According to this reference, levels 2 and 3 are valid, but level 3 has the point (V,S) shifted slightly to the right.

The four figures show that yielding follows the coulomb failure criterion, which has a form:

$$\tau_f = c + \sigma_n \tan \phi.$$

where τ_f is the shear stress at failure, c is the cohesion, σ_n is the normal stress, and ϕ is the internal friction angle. If we multiply both sides by the area A , the above equation becomes:

$$S = Ac + V \tan \phi.$$

For each case, the cohesion and the internal friction angle are calculated and shown in the appropriate figure. The combined results are shown in Figure 2.12, where:

$$\begin{aligned}\phi &= 24.8^\circ \\ c &= 3.57 \text{ g/cm}^2\end{aligned}$$

If we compare the value of the internal friction angle, 24.8° which we obtain against the value of 25.5° for glass beads given in the reference 3, we conclude that our result is in agreement with the reference. Our material shows non-zero cohesion, which is most likely caused by the moisture in the material due to humid weather.

INSTANTANEOUS YIELD LOCUS

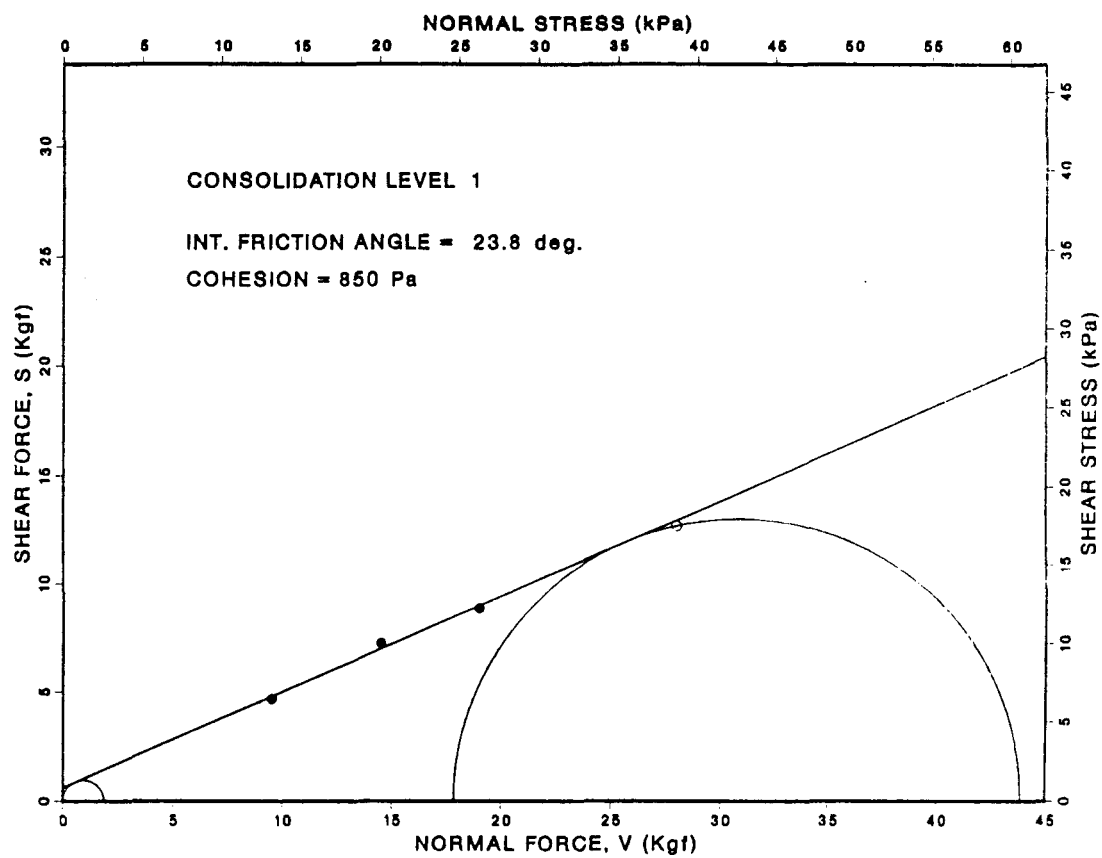


Figure 2.9 — Yield Locus of Level 1 Test.

INSTANTANEOUS YIELD LOCUS

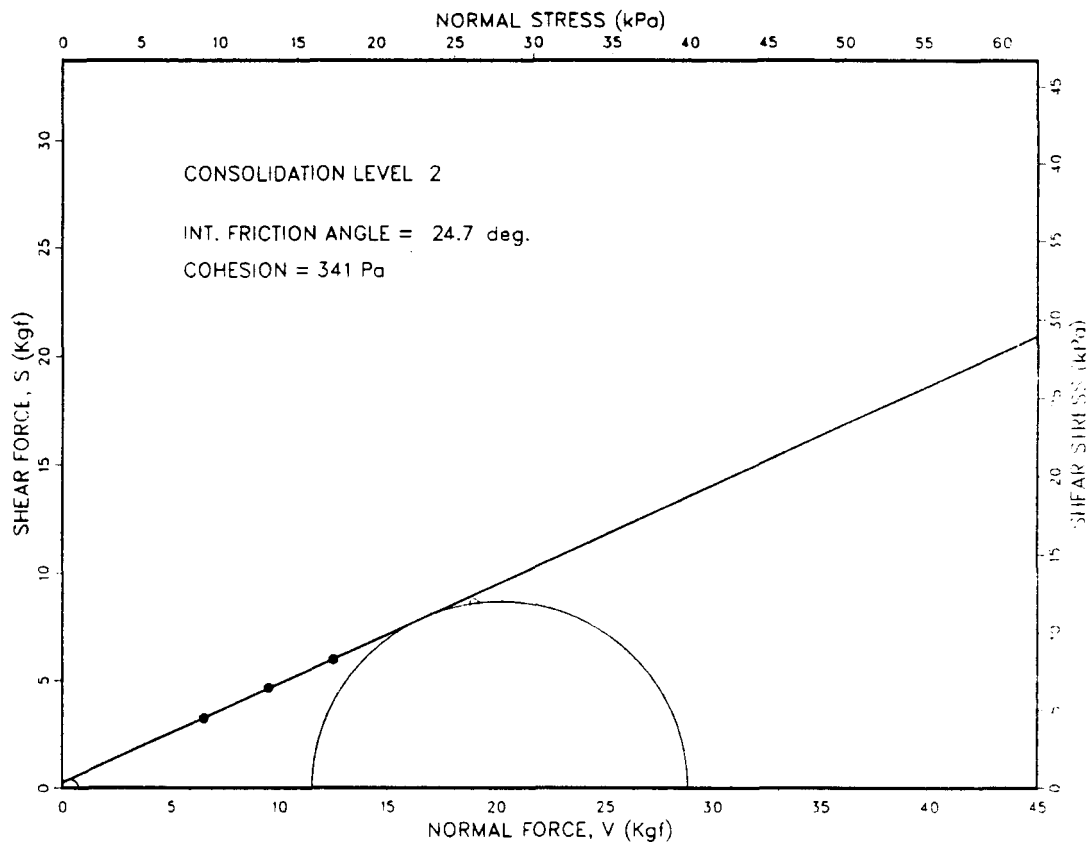


Figure 2.10 — Yield Locus of Level 2 Test.

INSTANTANEOUS YIELD LOCUS

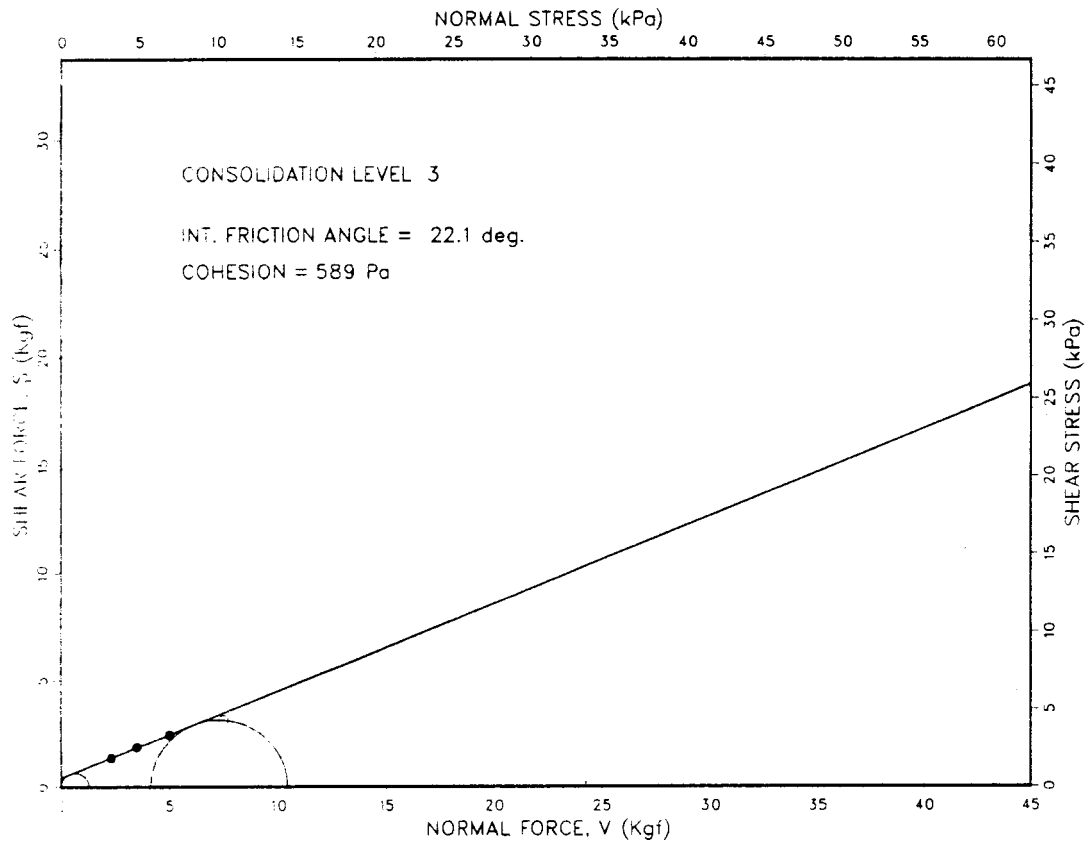


Figure 2.11 — Yield Locus of Level 3 Test.

INSTANTANEOUS YIELD LOCUS

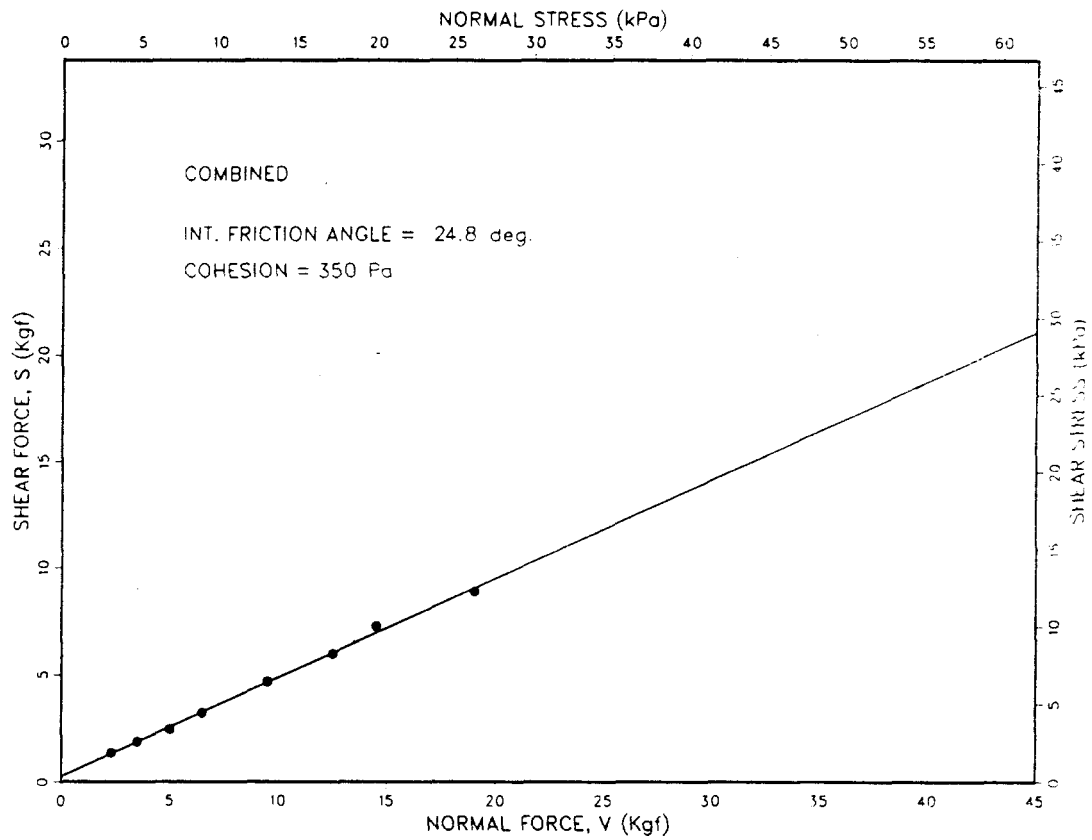


Figure 2.12 — Yield Locus of Combined Results.

3. REFERENCES

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2. Jenike, A. W., "Storage and Flow of Solids", Bulletin No. 123 of the Utah Engineering Experiment Station, University of Utah, Salt Lake City, Utah, 1964.
3. Brown, R. L. and Richards, J. C., "Principles of Powder Mechanics", Pergamon Press, 1970.
4. Savage, S. B. and Sayed, M., "Stresses Developed by Dry Cohesionless Granular Materials Sheared in an Annular Shear Cell", J. Fluid Mech., Vol 142 (1984), pp. 391-430.
5. Carr, J. F. and Walker, D. M., "An Annular Shear Cell for Granular Materials", Powder Technology, Vol 1 (1967/68), pp. 369-373.

A. APPENDIX A DYNAMIC SHEAR CELL TEST PROCEDURE

A.1 TEST CONDITION

Solids

Same as Jenike Shear Test

Environment

Weather: Mild
Room temperature: 23°C
Relative humidity: Not known

Recorder

Manufacturer: Hewlett Packard
Model: Mosley 7100 B strip chart recorder
Range: 10 in-lb/div.
Speed: .2 in/sec.

A.2 IDLE TEST I

The purpose of this test is to investigate the dynamic characteristics of the shear cell and to measure the friction force inherent in the shear cell. This is done without material in the gap.

Procedure

1. Turn the switches of the recorder on and wait 20 minutes for warm up.
2. Turn the motor controller on.
3. Assign the speed of the motor.
4. Place the pen of the recorder down and turn the advance switch on.

5. Press "RUN" at the bottom of the controller and then wait until repeated values are obtained.
6. Press "STOP".
7. Change the speed of the motor and then repeat from step No. 4.

B. APPENDIX B JENIKE SHEAR CELL EXPERIMENTAL CONDITIONS AND PROCEDURES

B.1 TEST CONDITIONS

Solids

Material: Glass beads (JAYGO Inc.)

Mean diameter: 80 μm

Condition: Stored in the test room.

Environment

Weather: Rain (93% relative humidity)

Room temp.: 23°C

Relative humidity in the room: Not known

Test Machine

Shear Test: FLOWFACTOR TESTER (Jenike & Johanson Inc.)

Model FT-35TEH

Stem speed = 2.7 mm/min.

Recorder: DYNAMASTER STRIP-CHART RECORDER SERIES 71A-550 (BRISTOL, Inc.)

Chart speed = 24 in/hr (10 mm/min.)

B.2 TEST PROCEDURE

1. Place the base, ring and mould on the disc of the consolidation bench with the offset of the ring of 2.5 mm.
2. Put the material in the cell gently, and scrape off excess material. Place the twisting top on the material.
3. Place the weight, W, (0.5 kg) on the weight hanger.
4. Rotate the twisting top with wrench while holding the ring and mold. The number of rotations and angle are determined in the pretests.

5. Remove the weight and carrier. Lift the mold upward and slide the twisting top off. Scrape the excess material above the ring.
6. Place the specimen on the disc of the shear unit.
7. Place the cover on the specimen.
8. Place the weight hanger on the cover, and then place weight W (0.5 kg), and steady the weight hanger.
9. Advance the stem turning the knob of the shear unit until the stem almost touches the bracket of the cover.
10. Place the switch in the advance motion position.
11. When the steady force is reached, place the switch in the retract position until the stem leaves the bracket.
12. Replace the weight by \bar{W}_1 (0.5 kg).
13. Place the switch in the advance position.
14. When the maximum shear force is passed, place the switch in the retract position.
15. Remove the weight.
16. Clean out the cell.
17. Repeat the test with different weight.

B.3 BULK DENSITY

Weighing device: Mettler PC 4400

Procedure:

1. Weigh the paper container with the scale, and measure the inside volume of the cell.
2. Place the cell on a clean aluminum plate.
3. Place the material in the cell and remove the excess material.
4. Clean the plate outside the cell.
5. Pour the material into the container.
6. Weigh the material and container

Weight of container = 5 g

Weight of the material and container = 183.1, 184.3, 175.5 g

Average weight of material $\Rightarrow 181.0 \text{ g}$
Bulk Density = $(181.0 - 5)/114 = 1.54 \text{ g/cm}^3$.

B.4 PRECONSOLIDATION

The optimum number of twists to consolidate the sample must be determined before shearing through the pretest. The twisting angle is specified as 30° .

Procedure:

1. Preconsolidation with specified number of twistings.
2. Consolidation test until the ring and base reaches the coaxial state.
3. Check the recorder output.
4. If a maximum force is observed before the steady state, repeat the test from 1 with fewer twists. If the recorded force does not reach the steady state, repeat the test from 1 with more twists.

Discussion:

1. Consolidation is sensitive to moisture content, vibration, and how to fill the cell up.
2. It is seen (from the decrease of the volume of material) that the packing fraction increases as a result of consolidation.
3. As consolidation weight decreases, the optimum number of twistings increases.

Table B.1 — Effect of Twistings

W (Kg)	twistings	state (value if steady)
27.5	<u>30</u>	13.1 Under 12.8 12.2
18.5	<u>30</u>	Under 9 Over 8.2 Under 8.5
7	30	Under Under Under
	40	Over Under
	<u>50</u>	3.4 3.15 3.2

Optimum twistings: 30 for W = 27.5 Kg
 30 for W = 18.5 Kg
 50 for W = 7 Kg