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Containment of a Silicone Fluid Free Surface in Reduced Gravity Using Barrier Coatings

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CONTAINMENT OF A SILICONE FLUID FREE SURFACE IN REDUCED GRAVITY USING BARRIER COATINGS

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SUMMARY

In support of the Surface Tension Driven Convection Experiment planned for flight aboard the Space Shuttle, tests were conducted under reduced gravity in the 2.2-sec Drop Tower and the 5.0-sec Zero-G facility at the NASA Lewis Research Center. The dynamics of controlling the test fluid, a 10-cSt viscosity silicone fluid in a low gravity environment were investigated using different container designs and barrier coatings. Three container edge designs were tested without a barrier coating: a square edge, a sharp edge with a 45° slope, and a saw-tooth edge. All three edge designs were success ful in containing the fluid below the edge. G-jitter experiments were made in scaled-down containers subjected to horizontal accelerations. The data showed that a barrier coating is effective in containing silicone fluid under g-levels up to 10^{-1} g_0. In addition, a second barrier coating was found which has similar anti-wetting characteristics and is also more durable.

INTRODUCTION

In the microgravity environment of space, due to the lack of bouyancy driven convection, materials processing involving solidification and crystal growth is expected to be dramatically improved. However, material properties will still be affected by thermocapillary flows induced by surface tension gradients. The Surface Tension Driven Convection Experiment (STDCE) (refs. 1 and 2) is planned as a microgravity flight experiment to study basic thermocapillary flow phenomena in a low gravity environment.

The STDCE (fig. 1) uses a 10 cm diameter cylindrical container (unit aspect ratio) filled with 10-cSt silicone fluid, and is planned to provide both a flat and a curved free surface which can be centrally heated either externally or internally. A planar cross section is illuminated by a sheet of light allowing observation of the resulting thermocapillary flows, generated by surface tension variation due to the temperature gradient along the free surface.

The success of a microgravity flight experiment depends heavily on how well the free surface is controlled. Silicone fluid has a very low surface energy (approximately 20 dynes/cm) and will wet (establish a near zero contact angle on) most surfaces. The free surface is contained by constant contact with the inner edge of the container. This is known as "pinning" the fluid to the edge. If the free surface is distorted enough by small accelerations called g-jitter, and causes

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the edge to be wet, fluid may begin to migrate out of the container, preventing the acquisition of fluid flow and temperature data from the desired geometry. If a substantial amount of fluid is lost and wets the remainder of the container it would be nearly impossible to retrieve the fluid without dismantling a significant portion of the experiment package, thus terminating the experiment.

In the literature (refs. 3 to 5) there are many papers dealing with lateral sloshing of fluids in tanks under zero-gravity conditions. This work describes the behavior of the free surface in sealed containers under a variety of conditions, but does not address the question of constant contact of the free surface with an edge inside the container (pinning). The effect of pinning on a moving fluid in low gravity is not well understood.

For this study a systematic test program was undertaken to determine the best way to pin the fluid at the container edge. In phase 1 of the test program, a series of drop tests was conducted in the 5.0-sec Zero-G Facility at the NASA Lewis Research Center to determine the best edge design for the container. Three designs were tested in a quiescent low gravity environment ($<10^{-5}\ g_0$) a square edge, a sharp edge with a 45° slope, and a saw-tooth edge (fig. 2). Each design was able to pin the surface at the inner edge.

In a parallel effort, a barrier coating (Scotchgard, a coating which will prevent the spreading of a fluid, i.e., possessing a lower surface energy) was found that could possibly contain silicone fluid. A series of drop tests was conducted in the 2.2-sec Drop Tower at NASA Lewis to verify the effectiveness of the barrier coating, applied to a container edge while subjected to horizontal accelerations.
Two edge designs were used: the square edge and the 45° sloped edge.

SYMBOL LIST

A amplitude

Bo axial Bond number

D container diameter

g_0 gravitational acceleration

g g-level

H container height

R container radius

V fluid volume

ρ density (silicone oil)

σ surface tension (silicone oil)

ω frequency

ω_n natural frequency

EXPERIMENTS

In phase 1 the three edge designs were tested to determine how well each edge pinned the fluid. A series of eight drop tests was conducted in the 5.0-sec Zero-G Facility. The facility is a 6.1-m diameter steel-walled vacuum chamber, extending 145 m into the ground. The chamber is evacuated to 1.3 Pa to minimize acceleration due to air drag ($<10^{-5}$ g_0 or 9.8×10^{-5} m/sec^2). The drop vehicle, a 1-m diameter by 1-m height cylinder, weighing approximately 1100 kg, is suspended at the top of the chamber by a notched bolt. When the bolt is sheared the package is released. The drop vehicle is decelerated by millions of polystyrene spheres in a large container at the bottom. The duration of the drop test is 5.2 sec (free fall distance is 132 m). The average deceleration upon impact is 35 g_0 (340 m/sec^2) for 120 ms. A schematic of the facility is shown in figure 3.

The test cell aboard the drop vehicle consisted of a 10.16 cm diameter plexiglas cylinder (approximately unit aspect ratio) with the appropriately machined edge. Before the drop test the cylindrical cell was filled with 10-cSt silicone fluid to the desired level. The drop vehicle was then closed up, moved to the top of the chamber, suspended and dropped. The motion of the free surface as it established an equilibrium shape was recorded with a high speed movie camera (400 frames/sec), and time marked with an LED timer in the camera field of view. The experiment was automated such that power was delivered to the camera and lights slightly before the vehicle was released. The timer began when the package was released. The film was retrieved from the drop vehicle, developed and viewed on a film analyzer. Between drop tests the plexiglas container was cleaned with alcohol and rinsed in a detergent and distilled water mixture. A schematic of the experiment package is shown in figure 4. The square edge was dropped twice and the other two edges three times each.

In phase 2 in the 2.2-sec Drop Tower, the effect on pinning of a barrier coating applied to the edge of the container was investigated. Unlike the previous pinning tests, most of these drop tests studied pinning under dynamic (g-jitter) conditions. This facility was chosen because of its low cost and ease of operation. The 2.2-sec Drop Tower provides 2.2 sec of low gravity by allowing an experiment package to free fall a distance of 27 m in a normal atmospheric environment (fig. 5). Air drag in this facility is minimized by allowing the experiment package to free fall inside a drag shield designed with a high weight to frontal area and a low drag coefficient. The only drag on the experiment package is the air drag associated with the relative motion of the package inside the drag shield. The resulting acceleration due to air drag on the experiment package is less than 10^{-5} g_0 (9.8×10^{-5} m/sec^2).

The experiment package and drag shield are suspended by a highly stressed music wire. The drop test begins when a pneumatic knife edge notches the wire, causing it to fail. The experiment and drop package are decelerated by a chamber of aerated silica sand causing a deceleration of about 20 g_0, which is controlled by varying the size and shape of the deceleration spikes mounted on the bottom of the drag shield (fig. 6). At the time of impact the experiment package has caught up to the drag shield and is resting against it. After the drop test the film is retrieved, developed and viewed on the film analyzer.

The experiment package consists of an oscillatory shaker table (variable amplitude and frequency) with a cylindrical plexiglas test cell (D = 3.175 cm, H = 3.81 cm) mounted on the table inside a plexiglas box to contain the fluid during impact. The container is filled to the desired level with 10-cSt silicone fluid. A high speed (128 frames/sec) 16 mm movie camera records the free surface motion. A schematic of the experiment package is shown in figure 7.

In order to provide a worst case of g-jitter it was decided to conduct these tests at the natural frequency of the fluid/container system. The natural frequency (ref. 6) given by

$$\omega_n = \left(\frac{2\pi\sigma}{\rho D^3}\right)^{1/2}$$

For a 10 cm diameter container the natural frequency is found to be 0.4 Hz. In order to see at least a few periods of this oscillation it would require more than 2.2 sec of free fall. Therefore the diameter of the container was chosen to be 3.175 cm. Therefore, the calculated natural frequency would be 2 Hz, allowing several periods to be seen during the drop test. The frequency range of the shaker table is 1.75 to 4 Hz and the natural frequency of the 3.175 cm diameter container is 2 Hz.

Since a fluid/container system is characterized by its axial Bond number, defined (ref 7) as the ratio of acceleration forces to capillary forces,

$$Bo = \frac{\rho g R^2}{\sigma}$$

a Bond number ratio between the two containers of unity should describe the same motion (ref. 6). If a Bond number ratio of unity is used, the ratio of the square of the diameters is inversely proportional to the ratio of g levels. Therefore extrapolating to the 10 cm diameter container

$$g_{10} = (0.1)g_{3.175}$$

the equivalent g-level to produce the same motion should be ten times less. The dimensionless g-level is equal to

$$g = \frac{A\omega^2}{g_0}$$

The results of phase 1 determined which edge designs would be used in phase 2. It was decided to use only the square edge and the 45° sloped edge for reasons that will be discussed below.

Eighteen drop tests were made in the 2.2-sec Drop Tower. As mentioned above, all but the first two were dynamic tests. The frequency used was very close to the natural frequency in most cases. The Scotchgard was purchased in its retail form (aerosol) with the following composition: 0.7 percent floroaliphatic resin (anti-wetting agent), 1,1,1-trichloroethane (solvent) and carbon dioxide (propellant). After each drop test the container was cleaned with asolvent (NA-500) which dissolves silicone fluid. The barrier coating was reapplied to the edge using a cotton swab after each cleaning. After applying the Scotchgard to the surface, a film of fluoroaliphatic resin was deposited when the solvent evaporated. The thickness of this film could not be measured, but was estimated, using a micrometer to be less than 0.003 cm.

RESULTS AND DISCUSSION

Phase 1

A summary of phase 1 results appears in table I. Each edge design showed an ability to pin the free surface to the edge. Of the eight drop tests conducted only three showed pinning. However, the failed drop tests were due to either an insufficient volume of fluid or spillage during handling before the test. In the five tests in which pinning did not occur, insight was gained into the behavior of the fluid in contact with a wetted edge. In each drop test the fluid starts from a flat free surface, slightly below the edge at time $t = 0$. After the start of the drop test ($t > 0$) the fluid rises up the container walls (to establish the lowest energy equilibrium free surface shape) until it reaches the edge and pins. The resulting free surface shape is curved.

Two drop tests were made with the square edge. Drop test number 2 had an insufficient amount of fluid (213 cc); therefore, 5.0 sec was not enough time for the fluid to reach the edge. The second drop test (no. 3: $V = 308$ cc) with the square edge, pinned the free surface successfully within 0.8 sec.

The 45° sloped edge was dropped three times. With $V = 308$ cc (no. 4) the free surface was pinned within 0.6 sec. The second two drop tests (no. 7: $V = 410$ cc, no. 8: $V = 400$ cc respectively) with the 45° edge were not successful because fluid was spilled during handling of the drop vehicle, thus wetting the edges, and no pinning resulted.

The saw-tooth edge showed an ability to pin when fluid was spilled. If fluid wet one tooth the next tooth pinned the free surface. This configuration was also dropped three times. The first drop test (no. 1: $V = 395$ cc) showed that the fluid was spilled over the first tooth during vehicle transfer. Although the first tooth was wetted, the free surface pinned on the second tooth. The third drop test (no. 6: $V = 410$ cc) was nearly identical with the exception that the free surface pinned to the third tooth. During the second drop test (no. 5: $V = 410$ cc) all edges were wet during vehicle transfer and no pinning was observed.

It is clear from these drop test results that all three edges were successful in pinning the free surface. The saw-tooth edge was a particularly practical design because it allowed for some spillage without sacrificing pinning. The second conclusion that can be drawn from these tests is that once the edge is wet, pinning is not likely to occur. The data from these tests are summarized in NASA Lewis' Motion Picture Department film "Surface Tension Convection Experiment, Zero-G Facility" (MPD no. 1691).

Phase 2

A summary of phase 2 results is shown in table II. The results of phase two are divided into three categories: (1) initial drop tests (2) barrier coating drop tests, which illustrate the ability of the barrier coating to resist the flow of silicone fluid and (3) anti-wetting drop tests, which illustrate that silicone fluid will bead up on coated surfaces. Figure 8 is a summary of test configurations showing edge design, coating, and initial and final surface shapes. Only two of the three edges tested in phase 1 were selected for phase 2 the 90° and the 45° edges. Because it was shown that all three designs worked equally well, the selection criterion was ease of manufacturing. The 45 and 90° edges were much easier to machine into the plexiglas container.

Drop test no. 1 (fig. (8a)) gave the first indication that this coating would be effective against silicone fluid in a microgravity environment. While trying to coat only the edge, small amounts of barrier film coated the inside wall of the container. During the drop test, the fluid spread up the side of the container and pinned at the coating on the inside of the container.

After seeing that the coating would halt the fluid even at this relatively high velocity (as opposed to a creeping flow over several hours in 1-g), the barrier coating was applied to the entire inside of the container (drop test no. 2, fig. (8b)). During this drop test the fluid spread up the side only enough to establish an equilibrium contact angle of roughly 40°, approximately the same as observed in 1-g.

The next seven drop tests (nos. 7, 4, 5, 8, 9, 15, and 16) were conducted to establish the effectiveness of the barrier coating to keep the free surface pinned under a horizontal g-jitter condition. Because fluid did not spread over the barrier coating in the first drop test, the barrier coating was used (without a solid edge) in drop test no. 7 to pin the fluid. The top 1 cm of the inside of the container was coated and the container was filled to within approximately 5 mm below this coating line (fig. 8(c)). The fluid spread along the wall until it reached the coating line, where it pinned. A g-level of 0.033 g_0 was applied without the fluid rising above the coating line.

In the remaining six drop tests (figs. 8(e), 8(d), 8(d), 8(d), 8(e), 8(d) respectively) the containers were fully filled and subjected to a variety of g-levels, from 0.013 to 0.1 g_0, by varying the amplitude and frequency of the shaker table, but keeping the frequencies close to the natural frequency. No spillage was observed during these drop tests. Extrapolating to the 10-cm-diameter container, an effective g-level of 0.0013 to 0.01 g_0 was experienced. The g-level required by the STDCE (ref. 1) is 10^{-4} g_0 to maintain a suitably quiescent free surface. These data indicate that in the event some g-jitter larger than 10^{-4} g_0 occurs (as much as one to two orders of magnitude), scientific data may be compromised but the worst case (fluid loss from the container) is minimized with the use of a barrier coating.

The final three drop tests (nos. 14, 17, and 18) were conducted under a high g-level (0.049, 0.049, and 0.072 g) to illustrate that even though fluid is spilled, repinning is not inhibited by wetting (figs. 8(e), 8(d), and 8(d) respectively). In drop test no. 14 some fluid spilled over the edge but left only a small drop beaded up on the edge, which does not affect pinning. Had there been no barrier coating, the entire edge would have been wet, inhibiting repinning. In drop test nos. 17 and 18 fluid also spilled over the edge of the container, but beaded up on the edge. Again, if there were no barrier coating, the fluid would have wet the entire edge, providing an opportunity for fluid to migrate from the container. A film summary of these results can be found in "The Effectiveness of a Barrier Coating for Microgravity Applications of Low Surface Energy Fluids" in NASA Lewis' Motion Picture Department (MPD no. 1710).

CONCLUDING REMARKS

The tests conducted were useful in visualizing the static and dynamic behavior of a free surface in contact with a container edge under reduced gravity conditions. It must be noted that there are several limitations to the interpretation of these data. First, the dynamic g-jitter tests conducted in the 2.2-sec Drop Tower are transient. Therefore, these data are most applicable to situations involving short term accelerations (i.e., jolts). Second, these tests address only accelerations parallel to the free surface. Accelerations normal to the free surface are a stability problem (refs. 8 to 11) and must be treated separately. Both parallel and normal g-jitter must be considered simultaneously in order to understand completely how pinning is affected during random accelerations.

There are also drawbacks involved with the surface flow inhibiting barrier coating. It is not very tolerant to abrasion. The resin can be wiped off a surface using light strokes with tissue paper. Also its effectiveness, when in contact with silicone fluid, is on the order of minutes. Since these tests have been conducted, a more appropriate (i.e., tougher and lower surface energy) barrier coating has been found (3M FC-723). FC-723 is a more appropriate barrier coating for the STDCE because it was designed for use with silicone products. It meets Military Specification MIL-B-81744A: Barrier coating solution, Lubricant migration deterrent. The surface energy of this coating is approximately 7 dyn/cm less than Scotchgard. 1-g tests indicate silicone fluid establishes approximately the same contact angle on FC-723 (placed on glass) as on Scotchgard. Its most important improvement over Scotchgard is its effective life on glass in contact with silicone fluid. It is at least 24 hours (per MIL-B-81744A) and tests indicate it could be as long as a month. Its life on glass, exposed to air, seems to be unlimited.

It must be noted that FC-723 has been used, unsuccessfully, previously in conjunction with the Drop dynamics Module aboard Spacelab-3 (ref. 12). Hypodermic needles were coated with FC-723 to restrict the creep of silicone fluid during drop injection of silicone fluid drops into an acoustic field. During the experiment, it was observed that silicone fluid did creep over the needles. The reason for the failure of FC-723 to contain silicone fluid was attributed to the effective life when in contact with silicone fluid. During pre-flight storage (approximately 4 months), silicone fluid creeped from the storage reservoir and came in contact with the FC-723, thus rendering it ineffective. Therefore, it is essential to ensure, through proper design, that silicone fluid will not, under any circumstances, come in contact with silicone fluid before the execution of the experiment.

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TABLE I. - SUMMARY OF PHASE 1 RESULTS

[90 = 90 Degree edge; 45 = 45 degree edge; ST = saw tooth edge.]

Drop number	Edge design	Volume, cc	Remarks
2	90	213	Volume of fluid insufficient to wet edge.
3	90	308	Completely pinned within 0.8 sec.
4	45	308	Completely pinned within 0.7 sec.
7	45	410	Surfaces wetted prior to drop. Inconclusive.
8	45	400	Surfaces wetted prior to drop. No pinning observed.
1	ST	395	First tooth wetted prior to drop. Pinning observed on second tooth.
5	ST	410	All teeth wet prior to drop. No pinning observed.
6	ST	410	First two teeth wetted prior to drop. Pinning at third edge.

TABLE II. - SUMMARY OF PHASE 2 RESULTS

[90 = 90 Degree edge; 45 = 45 degree edge; T = top edge of container; A = entire container coated; P = partial coating; F = filled to the edge; U = filled under coating.]

Drop number	Condition (edge-coating-fill)	Amplitude, cm	Frequency, Hz	G-level, g	Remarks
1	90-T-U	0	0	0	Fluid pinned at edge. Does not cover spilled barrier coating.
2	90-A-U	0	0	0	Fluid does not reach edge. Establishes approximately a 40° contact angle.
7	90-P-U	0.056	3.85	0.033	Fluid pins at barrier coating line.
4	45-T-F	.056	2.38	.013	No spillage.
5	90-T-F	.056	2.38	.013	No spillage.
8	90-T-F	.056	1.96	.0083	No spillage.
9	90-T-F	.056	2.86	.018	No spillage.
14	45-T-F	.32	1.96	.049	One drop of fluid beads up on container edge.
15	45-T-F	.32	2.86	.10	Large surface motion. No spillage
16	90-T-F	.32	1.5	.029	No spillage
17	90-T-F	.32	1.96	.049	Fluid rises over edge and beads up but remains pinned.
18	90-T-F	.32	2.38	.072	Fluid rises over edge and beads up but remains pinned.

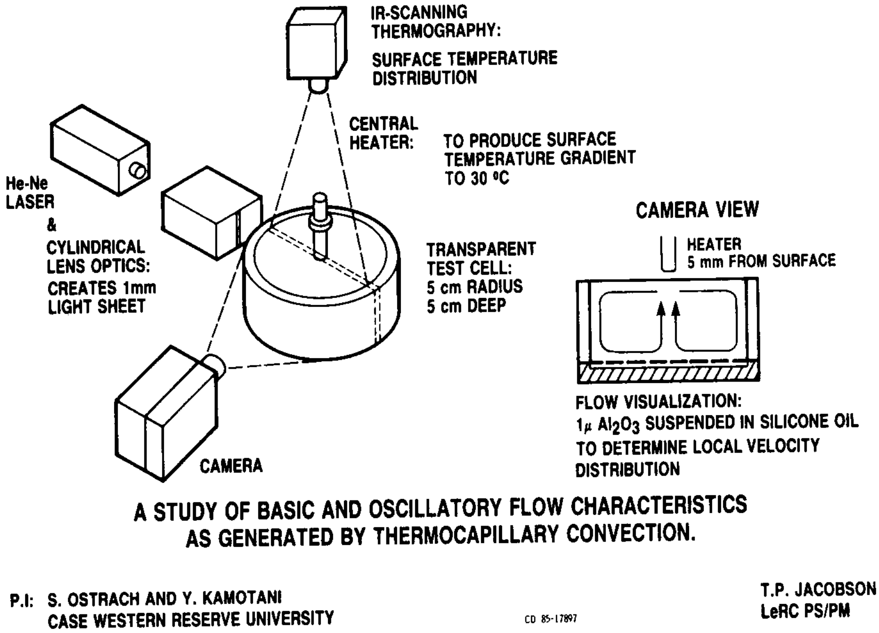

FIGURE 1. - SURFACE TENSION DRIVEN CONVECTION EXPERIMENT.

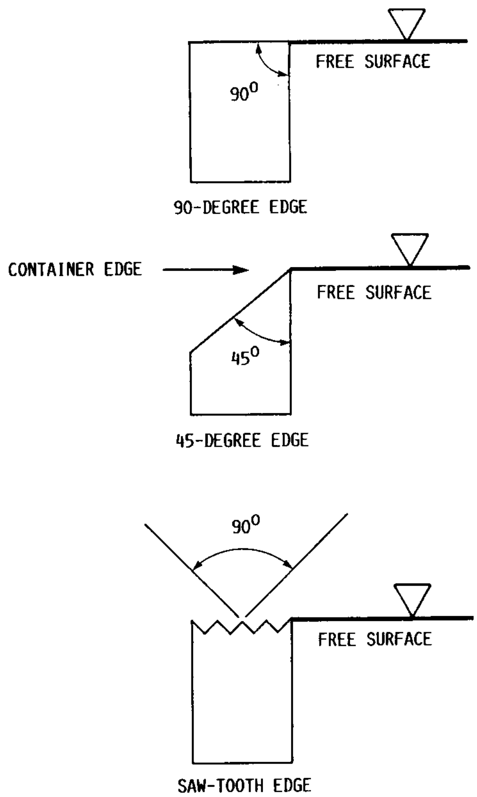

FIGURE 2. - EDGE DESIGNS.

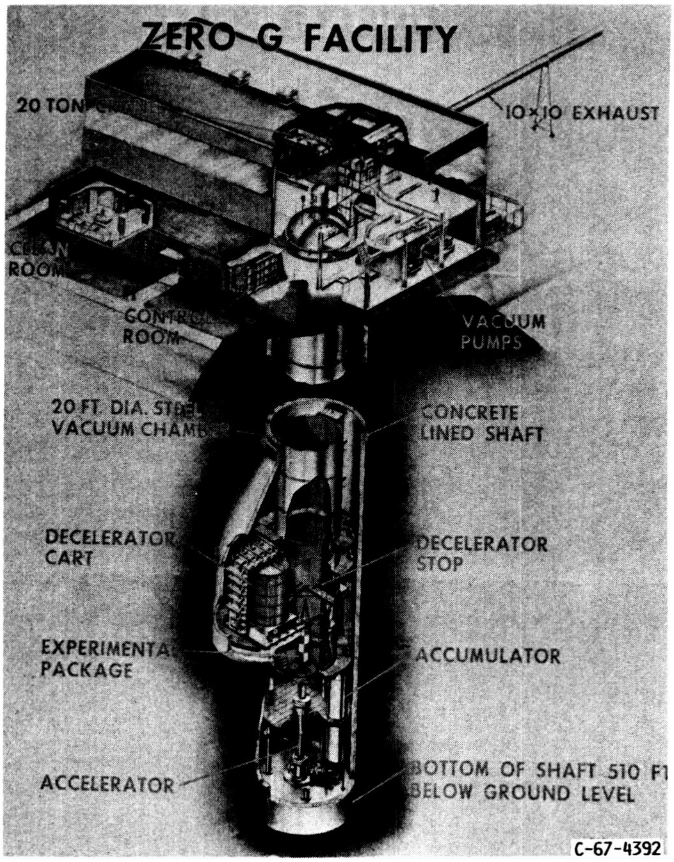

FIGURE 3. - ZERO-G FACILITY.

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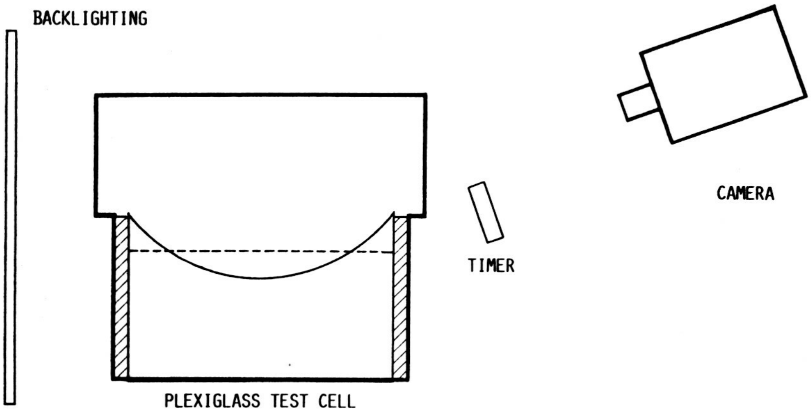

FIGURE 4. - SCHEMATIC OF EXPERIMENT PACKAGE USED IN PHASE I.

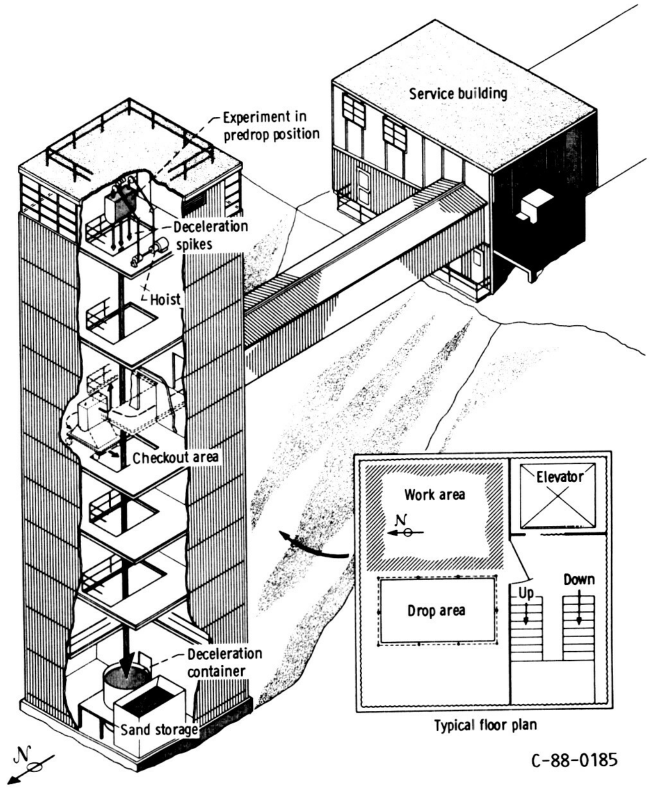

FIGURE 5. - 2.2 SECOND DROP TOWER.

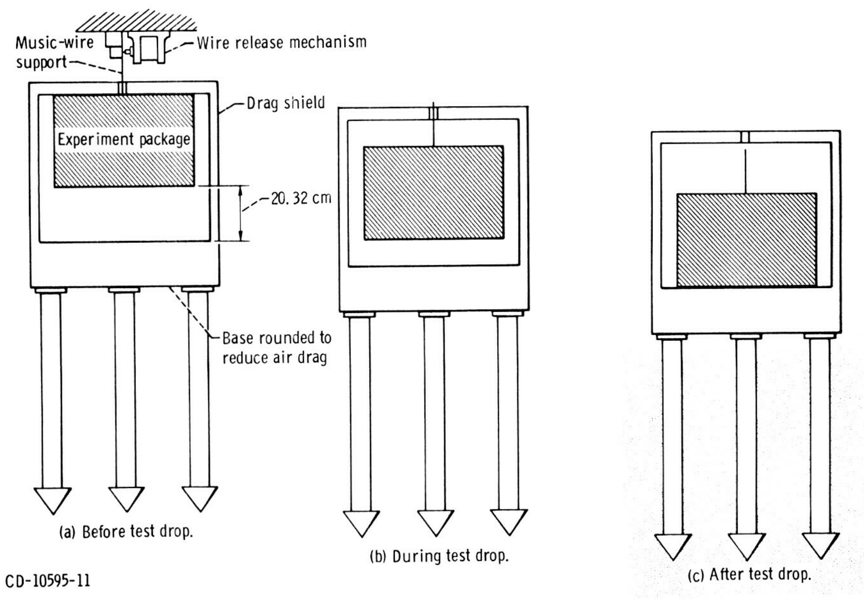

FIGURE 6. - DROP RIG AND EXPERIMENT PACKAGE.

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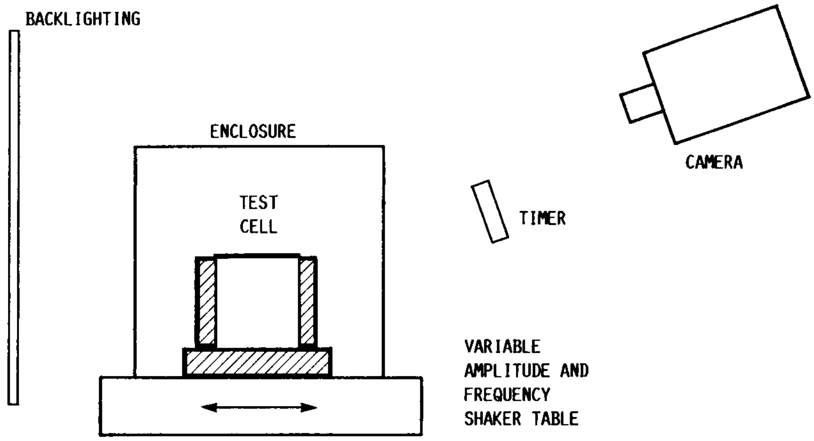

FIGURE 7. - SCHEMATIC OF EXPERIMENT PACKAGE USED IN PHASE II.

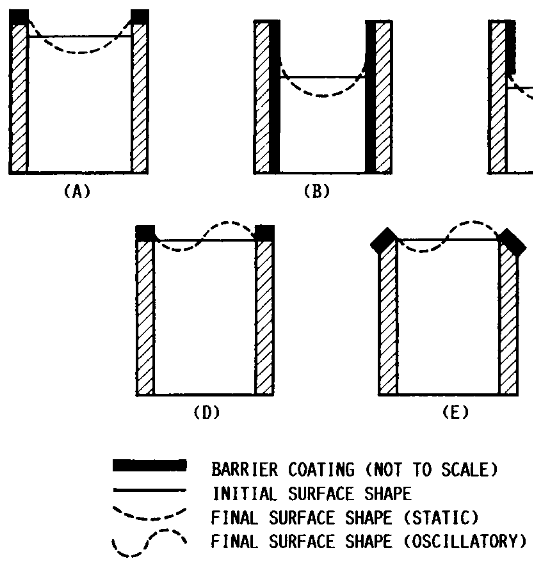

FIGURE 8. - SUMMARY OF TEST CONFIGURATIONS USED IN PHASE II.

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16. Abstract

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