DEVELOPMENT OF EQUIPMENT FOR

CLAY SWELLING TESTS

By

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Bachelor of Architectural Engineering

Oklahoma State University

Stillwater, Oklahoma

1961

Submitted to the faculty of the Graduate School of the Oklahoma State University in partial fulfillment of the requirements for the degree of MASTER OF ARCHITECTURAL ENGINEERING May, 1962

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Thesis Approved: Thesis Adviser adree Dean of the Graduate School

ACKNOWLEDGEMENTS

The writer could never express in mere words his deep appreciation to his advisor, Professor J. V. Parcher, for his valuable and untiring guidance, assistance, and encouragement. Gratitude is expressed also to Professor Parcher for his thorough editing of the final manuscript.

The writer wishes to thank Fred Gauger for the many hours devoted to preparing the excellent photographs contained herein.

Appreciation is also acknowledged to Robert Hawk for his friendship and timely assistance, especially during the final steps of preparation of this manuscript.

The writer also thanks Dr. C. A. Dunn, Executive Director of the Engineering Research and Experiment Station, who made available the funds and equipment for this research project.

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CHAPTER I

INTRODUCTION

General

It is not uncommon in the central-southwestern region of the United States for buildings to experience large amounts of damage due to volume change of the underlying clay deposits. This clay is commonly found to exist in various degrees of partial saturation. The relatively dry state of the clay is due to the semi-arid climatic conditions common to this area, characterized by long periods of drought intermingled with much shorter periods of moisture. With few exceptions, the surface clays of this region have experienced innumerable cycles of drying and wetting.

The desiccation of clays during a dry period may extend to depths of 15 to 20 feet. Shrinkage cracks break up the clay within this depth and may be as much as two or three inches wide. During the following wet period, water enters the cracks, causing the soil to swell.

Buildings erected during a period of drought or desiccation greatly retard the evaporative loss of water in the soil area beneath the structure. In fact, the clay will

absorb additional moisture by capillarity, which will be retained throughout the drying period, even though the evaporative water loss continues normally in the adjacent areas. Therefore, the interior portions of the building will heave by swelling with respect to the outer perimeter of the building. The weight of the floor slabs has practically no effect on the magnitude of the interior heaving. The heaving effect on the foundations of the building is similar to that of unequal settlement, except that the pattern is concave instead of convex.

Before an engineer can design a foundation in an intelligent and satisfactory manner, he must have a reasonably accurate conception of the physical properties of the underlying materials. This may require extensive investigation, as the strength, compressibility, and volume change capacity of the soils usually control the design.

One of the more important physical properties of soil in which the engineer is interested is the volume change caused by change in loading or in water content. When water becomes available to a precompressed soil, a volume increase occurs as the water is drawn into the void spaces. This increase in volume is generally referred to as swelling. If water is lost from the soil by evaporation, a volume decrease occurs until the tensile forces in the retreating water become maximum. This decrease is known as shrinkage, and the process of evaporation is referred to as desiccation. Increased loading produces a volume decrease by squeezing the water

from the void spaces and is termed consolidation.

The clay deposits found in this area are of such a nature that most differential settlements due to consolidation become relatively insignificant when compared to the differential settlements due to shrinkage or swelling. Thus the volume changes due to shrinkage and swelling of the clay under existing and proposed conditions and the maximum pressure intensities which may be exerted by the clay as it swells are of primary concern to the engineer in the design of structures for the central-southwestern part of the United States.

Purpose of Investigation

During the past several years, a great deal of effort has been put forth by the schools of architecture and civil engineering toward the discovery of an effective means of determining the extent of volume change which may be expected under various conditions (1 - 5). Other investigations have also been made in an attempt to reduce in magnitude the effects of volume change by swelling which are detrimental to structures (4).

A review of the previous swelling investigations has shown that they have been limited almost entirely to the study of vertical or one-dimensional expansion. A qualitative evaluation of the previously obtained data has been hampered by the limitations inherent in the particular type of apparatus used to obtain the data and by the detrimental variables common to most laboratory investigations.

It is felt that further investigations into the volume change of soils are certainly warranted, as the volume change characteristics of these regional clays still present a major problem in the construction of buildings and highways. These studies, however, should be extended to include not only the vertical expansion, but lateral and triaxial expansion as well. Studies of this nature are usually conducted using the standard triaxial shear strength apparatus.

A new apparatus was desired in preference to the types previously used (e.g., the consolidation apparatus, the triaxial apparatus, and others) in order to reduce the variables and limitations introduced by the particular apparatus. Consequently, the design and development of apparatus to be used in the determination of the swelling characteristics of soils is the primary purpose of this investigation.

It is anticipated that the end results of this endeavor will enable future investigations into the volume change characteristics of soils to be performed with a higher degree of accuracy, which will, in turn, facilitate the qualitative evaluation of data obtained.

Scope of Investigation

During the preliminary phases of this investigation, the object of prime importance was the swelling test apparatus and its development. The membrane to be used in conjunction with this apparatus was considered to be of lesser importance. It was presumed that a latex rubber membrane, as commonly used in the standard triaxial test, would be sufficient. The only reason the membrane entered into consideration early in the investigation was because the swelling apparatus designed required a flanged membrane rather than the straight-walled membrane previously used. Thus, the intent of the investigation was solely to develop a satisfactory apparatus and determine its performance characteristics during the proposed swelling tests. Little consideration was given to the possibility that the membrane might prove to be unsatisfactory.

It became evident at the completion of the first of the proposed swelling tests, for reasons discussed in Chapter III, that the order of investigative importance should be reversed. Therefore, the main investigative effort was diverted from the development of the testing apparatus to the development of an improved membrane to be used not only in this new apparatus, but also in the standard triaxial apparatus. This area of study ranges from the manner in which the membrane is formed to the physical properties of the membrane material under investigation. The data obtained are then compared to similar data pertaining to the latex membrane, where such data are available. The widely used latex membrane is known to possess some undesirable physical properties (e.g., excessive permeability over long periods of time) and a few unfavorable features occur in the forming process of the membrane. It is believed that an improved membrane would be a

very substantial contribution toward increasing the reliability of data obtained during the performance of various types of cylindrical compression tests.

CHAPTER II

SWELLING TEST APPARATUS

Basic Concepts

The original purpose of this investigation was the design and development of an apparatus with which the swelling characteristics of soils, compacted at various moisture contents, could be observed, tested, and measured. This apparatus was to be constructed in such a manner as to help eliminate some of the unfavorable characteristics inherent in currently used testing devices.

The consolidation apparatus, not unlike the apparatus developed by Alpan (6), is limited to the study of onedimensional (vertical) expansion studies. Neither of these apparatus allows the lateral confining pressure to be predetermined or varied for different tests. Alpan's apparatus, however, does provide for the measurement of the amount of water absorbed by the specimen at any time during the period of a test.

The standard triaxial testing apparatus was objectionable, due to the laborious procedure required for volume change studies. Direct measurement of lateral swelling is

possible, but rather inconvenient, with the use of an extensometer which is placed around the specimen, inside the The indirect method of lateral swelling measurechamber. ment requires the use of exterior equipment (i.e., burettes) which measures the amount of fluid forced out of the chamber by the swelling of the specimen. This method is cumbersome due to the number of connections and the small diameter tubing used between connections. It also yields rather inaccurate data as the ratio of confining fluid volume to sample volume is rather large and the exposed small diameter tubing is extremely vulnerable to the temperature changes commonly experienced in most laboratories. Another unfavorable aspect presented by the indirect method is the effect which vertical swelling has upon lateral swelling measurement during a triaxial study.

Portability of the apparatus, not available with the previously mentioned devices, also was desired as there are constant-temperature rooms available on the campus for laboratory use.

Upon the completion of what seemed to be a satisfactory testing apparatus, it was seen that a new type of membrane forming mandrel was needed. The previously used forming mandrel was a bullet-nosed cylinder of glass whose outside diameter was slightly smaller than the outside diameter of the soil sample. The new apparatus was designed for the use of a membrane with a flange at the top which holds the confining fluid inside the chamber during a vertical or triaxial expansion test. The most desirable shape was a membrane with a flange attached at right angles to the cylindrical body. This shape seemed inconvenient to manufacture since the only membrane forming method heretofore used consisted of dipping the mandrel into liquid latex which dripped dry and then was peeled from the mandrel. The shape shown in Figure 1 thus was deemed the most desirable due both to its ability to drain after being dipped and to its shape, which was satisfactory for the new apparatus.



Figure 1

Section Through Flanged Membrane Forming Mandrel

Description of Apparatus

nvenient *

In its final design form, the assembled apparatus with its appurtenances is shown in Plate I. The additional equipment shown is to be used for only the lateral expansion studies. The working drawings are given in Figures 2, 3, 4, and 5. Figure 6 is a schematic illustration of the apparatus









Figure 3

BASE DIATE	SOIL RECHANICE LABORATORY	SHEET
Scale . I ST	OKLAHOMA STATE UNIVERSITY	4
SWELLING TEST APPARATUS	125 7, 1962	4











Figure 6

Apparatus Assembled for Triaxial Swelling Test assembled for a triaxial (free) swelling test. The entire apparatus, excluding the fittings and needlepoint-micrometer water level gage, is constructed of lucite.

There are two sets of accessories used in conjunction with this apparatus. Set No. 1 may be used for either the vertical or the triaxial expansion studies, while set No. 2 is used for lateral expansion studies only. Each set consists of a cap and a cover plate, as shown in Figures 7 and 8.

The chamber, firmly clamped between the top and base plates with three tie rods, is tightly sealed at both ends using a standard O-ring connection in the bottom of the one-sixteenth inch groove which centers the plates over the chamber (see Figure 2).

To effect vertical confinement of the specimen, the thickness of cap No. 2 is such that, when placed on top of the specimen which in turn is mounted on the pedestal, the top of the cap extends exactly to the top of the top plate. The vertical expansion of the specimen is prevented by the use of cover plate No. 2, which is attached firmly to the top plate with screws. The uppermost seal which confines the chamber fluid is formed with another standard O-ring connection between the top plate and cover plate (see Figures 2 and 6). Cap No. 2 extends into the chamber for the purpose of binding the membrane to the cap, as the flanged membrane is not necessary for this type of investigation. However, if it is undesirable to bind the membrane to the cap, the flanged membrane may be used if a shim whose









Figure 8

Upper Connection for Lateral Expansion Study

thickness is equal to that of the flanged portion of the membrane is placed between the cap and cover plate. The shim will fill the space created by placing the flange of the membrane between the cover plate and the top plate.

If vertical expansion is desired, cap and cover plate No. 1 must be used. The hole in this cover plate requires the use of the flanged membrane in order to confine the chamber fluid during the test. The thickness of cap No. 1 is greater than that of cap No. 2 in order for it to extend into the hole in the cover plate, thus insuring the vertical movement of the cap through the cover plate during the initial stages of vertical swelling.

The water supply is directly connected to the specimen through the pedestal, which is recessed to contain a porous stone, and the base plate. The inside surface of the water supply reservoir is milled to a known diameter. Thus, using the needlepoint-micrometer water level gage, the amount of water which has entered the specimen can be accurately determined at any time during the test. A gasket is used between the gage and the reservoir to retard water evaporation.

The chamber fluid is connected through the base plate to a section of saran tubing. This tubing is affixed to the edge of a meter stick, which is attached to the apparatus at the top plate by means of a clip inserted under a tie rod wing nut. The stick is mounted at this level of the apparatus in order to maintain a slightly greater fluid level in the tubing than that which exists in: (a) the water supply

reservoir--to prevent the water flowing into the specimen from bulging the membrane, and (b) the chamber--to prevent gravitational flow of the confining fluid into the tubing. Since the inside diameter of the tubing is known and is reasonably uniform, any lateral swelling of the specimen can be accurately determined by measuring the displacement of the liquid-air interface within the saran tube.

A valued connection is the only connection which is made through the top plate into the chamber. Once the apparatus is assembled for the type of test desired, the chamber is filled with the confining fluid through the base plate chamber connection until the fluid is forced through the valued connection into an attached bleeder line. The function of the bleeder line is to provide a reservoir of the chamber fluid which may be caused to flow back through the base plate connection into the saran tubing, which is connected after the chamber is filled and sealed.

Recommended Procedures

Preparation of Specimen

The preparation recommended by Lambe (7) may be followed in obtaining an undisturbed specimen. The only deviation from his procedure is in the length of the specimen, which should be 2.8 inches.

Remolded specimens, compacted at various moisture contents, may be prepared as follows:

1. Thoroughly mix approximately 140 grams of soil with

the amount of water required for the water content desired.

Store the mixture in a humid room for approximately
24 hours. This allows the water to become more uniformly
distributed throughout the soil.

3. After compacting in the Harvard Miniature Compaction Apparatus, modified to produce a specimen 1.4 inches in diameter and 2.8 inches long, wrap the specimen in metal foil, seal with wax, and store in the humid room until ready for testing. (Specimens prepared in this manner can be stored for many months without appreciable change in moisture content.) A sealed specimen is shown in Plate II-1.

4. The trimmings from the compacted specimen are used for moisture content determination.

Swelling Tests

After removing the wax and foil, enclose the specimen within the membrane by using a membrane stretcher, as shown in Plate II-1. Weigh the specimen and the membrane.
(A straight-walled membrane is used for lateral swelling tests, while a flanged membrane is used for the vertical or triaxial swelling tests.)

2. Deair the pedestal, base, and connections between the base and reservoir by flushing with distilled water. Place a porous stone, which has been previously deaired, in the recess provided in the top of the pedestal.

3. While the distilled water is still flowing from the reservoir through the porous stone, close valve (a). Remove the excess water standing on the porous stone and place the



1. Placing Specimen in Membrane



2. Installing Lower O-ring Membrane Seal



3. Installing Upper O-ring Membrane Seal



4. Completed Assembly



APPARATUS ASSEMBLY PROCEDURE - LATERAL EXPANSION STUDY

specimen atop the pedestal, lapping the end of the membrane over the pedestal. Thus, practically no water will be absorbed by the specimen until valve (a) is reopened.

4. Place the cap on top of the specimen, then lap the upper end of the membrane over the cap. (Use the cap required for the desired test.)

5. Using the metal O-ring expander to expand and pass the O-rings down around the specimen, place two O-rings over the bottom end of the membrane which overlap the pedestal, as shown in Plate II-2. Care should be taken to prevent membrane wrinkles under the O-ring, more especially, a wrinkle which extends under both O-rings. Some time should be spent prior to an actual test in practicing this step, as the correction of careless placement of an O-ring may be difficult.

For Lateral Swelling Tests:

5(a). Two O-rings are also placed over the membrane enclosed cap using the same procedure as in step (5), as shown in Plate II-3. These two O-rings must be placed within one-half inch of the bottom of the cap in order for the top plate to be properly positioned.

For All Tests:

6. Place the chamber sealing O-rings into position. Set the chamber into the centering groove in the base plate. Using the centering grooves as a guide, center the top plate on the chamber, taking care not to jostle the cap as the top plate passes down around the cap. If a flanged membrane is

used, the flange should be lying on top of the top plate upon completion of this step.

7. Insert the three vertical tie rods through the top plate and screw them into the base plate until finger tight. Next, after placing the meter stick hook over one of the rods, tighten all wing nuts until finger tight. Place the uppermost O-ring in place on top of the top plate.

For Lateral Swelling Test:

8(a). Screw cover plate No. 2 onto the top plate. Do not tighten screws.

For Vertical or Triaxial Tests:

8(b). Press the membrane flange to the top plate with cover plate No. 1 and puncture the flange by inserting a nail through the screw holes. Loosely screw cover plate No. 1 to the top plate.

For All Tests:

9. Fill the chamber with the confining fluid through the base plate chamber connection until the fluid, after sufficiently filling the bleeder line, starts to seep from under the cover plate. Then tighten the cover plate screws one-half turn at a time until the O-ring seal is effected.

10. Remove the confining fluid supply line from the base plate chamber connection and install the saran tubing connection. Adjust the liquid-air interface within the saran tube by elevating the bleeder line above the meter stick to which the saran tube is attached. When the desired initial position of the interface is attained, close valve (b). 11. If lateral pressure is desired, apply air pressure through the saran tubing, subjecting the entire chamber fluid system to the same pressure. If the air pressure supply reservoir is sufficiently large, the movement of the liquid-air interface will not be appreciably inhibited.

12. Add distilled water to the supply reservoir until it is about three-fourths full. With the needlepointmicrometer water level gage affixed tightly over the reservoir, determine the initial level of the water.

13. Take the initial chamber fluid reading on the meter stick.

For Vertical and Triaxial Tests:

14. Tightly screw the dial holder rods into the top plate and install the dial holder and the dial gage for measuring vertical deflection. Adjust the dial holder until the plunger is in contact with the cap. Take the initial dial reading.

For All Tests:

15. Open valve (a) to start the test.

16. Take readings of reservoir water level, vertical expansion (if tested), displaced chamber fluid, and time.

17. If lateral pressure is used for test, make sure it remains constant during the test period.

18. Continue to take readings until the completion of the test (indicated by no further expansion of specimen).

19. Drain the chamber fluid after closing open valves. This is done through the base plate chamber connection. Disassemble the apparatus.

20. Weigh the membrane enclosed specimen. The difference between this weight and that obtained in step (1) is the amount of water absorbed by the specimen and should check closely with the amount determined using the needlepointmicrometer water level gage.

Swelling Pressure Tests

If the swelling pressure, rather than the swelling expansion, of the specimen is desired, a pressure gage may be installed at valve (b) to measure the lateral pressure and a proving ring installed in place of the vertical expansion dial to measure the vertical pressure. If preferred, loads can be applied directly to the cap until vertical movement is prevented, thus indicating the vertical pressure exerted by the specimen.

CHAPTER III

MEMBRANE DEVELOPMENT

Basic Concepts

The length of time required to complete one of the swelling characteristic studies presented in the preceding chapter will vary from several days to several weeks, depending upon the physical properties of the specimen. Lambe has stated, "Although water is satisfactory for tests of short duration--e.g., a couple of days or less--it is not recommended for longer tests because it tends to permeate the membrane."

For long term tests, the soil testing laboratory at another university was known to have had reasonable success using silicone oil as chamber fluid in conjunction with natural rubber latex membranes. With this fact in mind, silicone oil (Dow Corning 702 Fluid) was used as the chamber fluid in the triaxial swelling test discussed in Chapter IV. However, at the conclusion of the test, the latex membrane was found to be badly weakened and deteriorated by this particular type of silicone oil.

The above performance and the preferred use of water

for expansion study tests, due to its availability and convenience in handling, meant that a less permeable membrane would be desirable.

Ideally, the membrane and the material from which it is manufactured would conform to the following specifications:

1. Material easily applied to forming mandrel.

2. No material wastage during application.

3. No osmosis through the membrane, i.e., impermeable.

4. Perfect elastic behavior of membrane with rapid deformation.

5. Recoverable "creep" action of membrane with slow deformation.

6. No weight or volume change after immersion in distilled water.

7. High tensile strength.

8. Low extension modulus.

After considering the problem at hand, it was felt that silicone rubber would be the material best suited for the purpose of obtaining a satisfactory membrane. Specifically, the material selected for this investigation was Room Temperature Vulcanizing (RTV) Silicone Rubber produced by General Electric.

Presently, six RTV Silicone Rubber compounds are offered by General Electric. Each compound is identified by a number which serves to indicate its relative viscosity or flow characteristics in the uncured state, which is the major difference between each compound. Thus, the lower the number assigned to a compound, the lower or more fluid is its viscosity. Table I illustrates the identification of RTV compounds.

TABLE I

Viscosity, Compound Color Poises Consistency RTV-11 White 120 Easily Pourable RTV-20 Pink 300 Pourable **RTV-40** White 450 Pourable **RTV-60** Red 550 Pourable 10,000 **RTV-88** Red Spreadable Thixotropic Paste **RTV-90** Red 12,000 Stiff Paste

IDENTIFICATION OF RTV COMPOUNDS

A general description of the physical properties of RTV Silicone Rubber is given in Table II. Table III gives the volume change properties of RTV-60 after immersion in water, or in salt water. It should be noted that the properties listed are "typical values" only.

Tables I-III were compiled from various General Electric publications, primarily the "S-3 Data Book," and from personal correspondence. Only the data pertinent to this investigation is presented.

The cure characteristics of any RTV compound can be controlled by: (1) the type of curing agent (catalyst) used-e.g., Thermolite-12 catalyst provides a relatively fast cure,
TABLE II

Compound	Specific Gravity	Durometer Hardness, Shore A	Tensil e Strength, psi	Elongation $\%$	Shrinkage %
RTV-11	1.18	45	350	180	0.5
RTV-20	1.30	50	450	140	0.2
RTV-40	1.37	55	550	120	0.2
RTV-60	1.45	60	650	110	0.2
RTV-88	1.48	65	700	110	0.5
RTV-90	1.45	60	750	160	0.2

PHYSICAL PROPERTIES OF CURED RTV

TABLE III

PER CENT VOLUME CHANGE OF RTV-60 AFTER IMMERSION

		Solution Used	for Immersion	
	Water		3% Aqeous NaCl	5% Aqeous NaCl
3 Days/1580 F.		4 Days/212 ⁰ F.	2 Days/158 ⁰ F.	3 Days/80° F.
3%		7.4%	- 0.6%	1%

while Silicure T-773 gives an extra fast cure; (2) the quantity of catalyst used--i.e., increasing the amount of catalyst will increase the rate of cure and decrease the pot life and vice versa; and (3) the temperature applied during the cure-i.e., the application of heat to a catalyzed compound will decrease the tack free time and firm time (rate of cure), but will also decrease the pot life or working time.

The special terms used above are defined for the layman in the "S-3 Data Book" as follows:

1. Pot life is the length of time between catalyst addition and cessation of compound flow.

2. Tack free time is the time after which all surface tack is eliminated.

3. Firm time is the time required to obtain about 40 Shore A durometer hardness on a one-half inch thick sample.

Only RTV-60 can presently be obtained in either the "bulk" compound or the aerosol spray form. In spray form, RTV-60 retains all of the desirable properties of conventional liquid silicone rubber previously described. It does differ, however, in application procedure, cure time, and handling instructions.

The spray should be applied perpendicularly to, and at a distance of six to ten inches from, the sprayed surface. Three complete coverage passes will provide a thickness of approximately four mils without runs. The **RTV** Catalyst Spray, used in conjunction with the **RTV-60** spray, should be sprayed from a farther distance. Spraying too closely tends to wash the RTV-60 from the sprayed surface.

Manufacturing Attempts

As previously stated, the membrane to be used in conjunction with the developed apparatus must be either flanged, for vertical or triaxial expansion, or straight, for lateral expansion. Thus, the shape of the membrane is governed by the configuration of the test apparatus and the type of test to be performed.

There are no rigorous requirements for the thickness of the membrane. However, it is necessary for the membrane to be of such thickness as to produce the optimum combination of the following: impermeability, expansibility, and strength. For tests whose duration does not exceed one or two days, this optimum is achieved in the latex membrane with a thickness of approximately eight mils. For long term tests, however, a greater thickness is necessary in order to obtain sufficient impermeability. However, due to the restraint furnished to compression and expansion of the soil, the thick membranes require the use of a correction factor in the evaluation of the test data.

Since the RTV rubber was believed to be more impermeable than latex, the efforts described in the following sections were directed toward obtaining a manufacturing procedure which would consistently produce a membrane of approximately six mils in thickness. Also, this thickness was desired in order to eliminate the use of a correction factor to compensate for membrane restraint of the soil.

RTV-60

Trial No. 1--Brush application of bulk RTV-60 catalyzed with two per cent Thermolite-12. No release agent was used for this trial.

The catalyzed compound was found to be too stiff to flow into a uniform thickness after being applied by brush. Plate III-1 illustrates the grooves and irregular surface texture caused by the brush bristles. After curing, an attempt was made to peel the RTV from the mandrel. This was found to be futile since the RTV had adhered to the flange of the mandrel, particularly in the minute grooves. These grooves are the result of the manner used to form the flange of the mandrel--i.e., expansion of a soft glass cylinder. However, it was possible to peel the RTV from the smooth cylindrical trunk of the mandrel, except for small pinpoints which stuck to the mandrel. These pinpoints made it impossible to obtain an intact membrane, as no warning sign preceded their appearance during the peeling process.

From this trial, it was concluded that a release agent was necessary if a membrane was to be obtained intact. Moreover, if brushed or dipped application is continued, an RTV compound of lower viscosity will be required in order to attain the texture and uniform thickness desired.

Trial No. 2--Spray application of RTV-60 Aerosol with Silicure T-773 Catalyst Spray.

From a preliminary application on a flat glass plate, it



1. Brushed on a Plate (Undesirable)



2. Sprayed on a Plate (Desirable)



3. Sprayed on a Mandrel ("Orange-peel")



4. Sprayed on a Mandrel (Satisfactory)

Plate III

RTV-60 TEXTURES OBTAINED ON STATIONARY FORMS

was noted that this type of application produced a desirable texture (see Plate III-2), but also required the use of a release agent, as the RTV-60 spray conforms to minute irregularities even more readily than the bulk RTV-60. Thus, release agents of silicone stopcock grease and Silicure T-773 Catalyst Spray were used on separate mandrels. Four complete coverage passes were applied to each mandrel. Plate III-3 illustrates the "orange peel" outer surface texture obtained on each mandrel.

The mandrel with the RTV Spray Catalyst as a release agent yielded a membrane which was very easily peeled from the mandrel. Evidently, the Spray Catalyst had filled the tiny grooves previously mentioned and covered any blemishes which existed on the surface of the mandrel.

The membrane formed on the mandrel with the stopcock grease as a release agent could be peeled from the mandrel, but it had a nonuniform thickness due to the hand application of the stopcock grease.

Trial No. 3--Spray application of RTV-60 with Silicure T-773 Catalyst Spray used as both catalyst and release agent.

Four mandrels received this application and yielded four usable membranes. Each membrane was of uniform thickness with a smooth inside surface texture. It was noted that the outside "orange peel" surface texture was present on only two of the membranes. The outside surface texture of the other membranes (see Plate III-4) closely resembled that previously obtained on the flat glass plate. Upon consulting the Silicone Products Department of General Electric, it was learned that the "orange-peel" surface obtained from RTV Aerosol Spray is a common problem. However, as evidenced by this trial, it is a matter of individual technique and sometimes can be eliminated. The "orange-peel" seems to be increased through the application of the spray from the incorrect distance or through the spray striking the surface at the wrong angle.

Further Trials with RTV-60

It was decided that rotating the mandrel during the spray application of RTV might facilitate the elimination of the "orange-peel" surface texture. With this purpose in mind, the mandrels (flangeless) were mounted on metal rods, as illustrated in Plate IV-1. By gripping the rod within the chucks of a hand drill which was connected to a rheostat, a mandrel could be rotated at any speed desired. Plate IV-2 illustrates the technique used for spray application onto a mandrel rotated within the spray booth constructed for this purpose.

An initial application was made on a flanged mandrel rotating at approximately 2100 r.p.m. This speed is attained when the drill is connected directly to the wall outlet. From the bumpy texture obtained, as shown in Plate VII-1, it would appear that this speed was the "critical" speed for the diameter of mandrel used--i.e., a higher speed would have thrown the bumps off of the mandrel, and a lower speed would



1. Mandrel Mounted on Rod



3. Dipping Flanged Mandrel



2. Spray Technique



4. Draining "Dipped" Flanged Mandrel

Plate IV RTV APPLICATION



1. Straight Walled Silicure T-773 Spray as Release Agent



2. Flanged - Polylease-77 as Release Agent

Plate V

PEELING THE MEMBRANE



1. Silicure T-773 Spray as Release Agent



2. Polylease-77 as Release Agent

Plate VI

INSIDE SURFACE TEXTURE OF RTV-60 MEMBRANE





Plate VII

RTV-60 TEXTURES OBTAINED ON ROTATING MANDRELS

not have caused the formation of the bumps.

A series of applications were made on mandrels which were sequentially rotated at speeds which ranged from 2000 r.p.m. to 400 r.p.m. Each mandrel was presprayed with spray catalyst to facilitate subsequent stripping of the membrane. After applying each of the numerous coatings necessary to attain the desired membrane thickness, visual observations were made and are recorded in Tables IV and V. Plate VII illustrates the textures obtained at the different speeds.

Table IV represents the first half of the application on mandrels rotating at speeds of 2000 r.p.m. to 1000 r.p.m. Each coating consisted of four sprayed layers of RTV-60. Spray catalyst was applied after each coating. Each coat was allowed to cure for approximately 45 minutes before the next coating was applied.

Since no further coatings were applied to the 2000 r.p.m. membrane, in order to obtain a thin membrane, Table V represents the second half of the application on mandrels rotating at speeds of 1800 r.p.m. to 1000 r.p.m. For this application, due to observations during the first half application, each coating consisted of four layers applied in pairs, with the spray catalyst applied to each coat.

The RTV-60 Aerosol Spray was applied to the 2000-1000 r.p.m. mandrels through a distance of approximately twelve inches. This spray distance was shortened to approximately eight inches for the 800-400 r.p.m. applications. Also, the 800-400 r.p.m. mandrels were allowed to cure only 15 minutes

TABLE IV

Coat No. 3 R.P.M. Coat No. 1 Coat No. 2 Coat No. 4 2000 Increased "orange-Much smoother with Slight "orange-peel," peel" texture All mandrels ap-(#1)similar to the very slight "orangestandstill texture peel" pear to have a 1800 More "orange-peel" Texture smoothed Very slight "orange-(#2)than #1 out, almost no peel" similar texture, "orange-peel," looks like #1 which, for all 1600 Slight "orange-peel" "Orange-peel" smooths Smoother texture practical pur-(#3)similar to #1, but than before out, becoming small smoother grained in texture poses, is smooth. Slightly smoother, Slight "orange-Same as before. 1400 The only irregupeel" similar to (#4)small grained, maybe smoother in uniform texture texture larity is due to #3 the spray and the Very slight "orange-Slightly roughened, 1200 Almost no "orange-(#5)peel," almost peel," catalyst increased "orangespray particles. made no difference peel," but less smooth than others Even this irregu-1000 Texture is the Still smooth, Roughened slightly, larity is barely (#6)but still the smoothest of all catalyst made no difference smoothest in texnoticeable. ture Addition of cat-Com-Texture is smoother Sprayed surface of ments alvst seems to after 2 passes than all mandrels be-

after 4 passes.

roughen texture.

coming uniform in

texture.

RTV-60 ROTATING APPLICATION--FIRST HALF

TABLE V

R.P.M.	*	Coat No. 5	Coat No. 6	Coat No. 7	Coat No. 8
1800 (#2)	1.	"Orange-peel" re- turned	Approximately same texture, still smooth	Smooth, even wavy texture	Used old spray can, "orange-peel" returned
	2.	Smoothed out, with shiny surface	Almost smooth again	Over-sprayed and "orange-peel returned	Very "orange-peel," but still shiny
1600 (#3)	1.	Texture roughened	Smoothed slightly, still fine grained	Fine grain texture changing to 'pigskin''	Used new can, still has most "pigskin"
	2.	Fine grained tex- ture returning	Stayed the same	Smoothed, but still greatest in "pigskin"	Smooth, shiny, slight "pigskin"
1400 (#4)	1.	Roughened, but still quite smooth	Very smooth	Stayed the same	Slightly roughened
("-)	2.	Very smooth, hard to see "orange-peel"	Stayed the same	Still smooth, some pores appearing	Slight "orange-peel" due to over-spray
1200 (#5)	1.	Same as #4	Same as #4	Stayed the same	Still slightly rough
-	2.	Stayed the same	Stayed the same	Starting to roughen, still smooth	Slightly rougher, similar to #4
1000 (#6)	1.	Same as #4	Stayed the same	Stayed the same	Irregularities more well-defined
	2.	Slightly rougher	Stayed the same	Same as #5, with a few pores	(Texture same for all 8 coats)
Com- ments		Using new spray can seems to cause sur- face to shine.	New can seems to spray out more par- ticles which roughen the texture.	Pores appearing in surface. These are referred to as "pigskin".	High gloss, regard- less of texture, on all membranes.

RTV-60 ROTATING APPLICATION--SECOND HALF

*This column denotes half-coatings--two layers each.

between coatings which were applied in pairs.

No table of observations is presented for the 800-400 r.p.m. mandrels as there was no visible difference between them, and this over-all appearance was not affected by the individual coatings throughout the entire forming process. It was noted that a quick coverage (one-half second per layer) produces a well-defined texture, while a slow coverage (one second per layer) yields a more gentle, rolling texture.

RTV-11

<u>Trial No. 1</u>--One mandrel, dipped into bulk **RTV**-ll and allowed to drain until dripping stopped; then Silicure T-773 Catalyst Spray was applied. The mandrel was presprayed with Silicure T-773 Catalyst Spray as a release agent.

Prior to the application of the spray, the surface of the membrane appeared smooth and uniform. Afterwards, the surface became irregular and the compound began to drip from the mandrel again. The mandrel was left in this position-i.e., tip down--for about an hour during which it was unobserved. When the mandrel was observed again, the surface of the membrane had become extremely irregular and folded. The majority of the compound had collected toward the nose of the mandrel and was covered with a thin shell of catalyzed **RTV**. The compound which remained near the flange seemed to be untouched by the catalyst, as it exhibited considerable surface tack approaching that of the original compound. At the time, no reason for this behavior could be postulated. <u>Trial No. 2</u>--The same procedure was used as in trial No. 1, except that the catalyst spray was applied very lightly and the mandrel was placed with the flange down during the cure.

The application of the spray did not seem to affect the surface of the membrane. In fact, the spray was too light to affect any amount of curing, as the **RTV** had not begun to lose its surface tack after a time lapse of a week. Also, placing the membrane in a 70° C. oven to effect the cure was attempted, but proved to be of no avail.

Trial No. 3--Application of RTV-11 catalyzed compound, using 2% Thermolite-12 as catalyst. The release agent used in this trial was aerosol polyethylene (Polylease-77 from Ren Plastics).

The compound was applied by brush onto two mandrels and poured over a third. The latter mandrel was rotated by hand to throw off the excess compound as it ran down the mandrel onto the flange.

The surface texture of each membrane was very smooth. The membrane formed by pouring was the most uniform in thickness. The membrane thicknesses obtained in this trial were much greater than desired. The viscosity of the compound will have to be modified in order to reduce the membrane thickness if this method of application is to be successful. It was noted that the membranes which were placed with the tip down during the cure were very irregular and lumpy throughout the flange area, as illustrated in Plate VIII-1. This flange thickness irregularity might possibly be eliminated by inverting the mandrel during the cure.



1. Flange Irregularities of Brushed Application



2. Dipped and Rotated at 80 r.p.m.



3. Brushed and Rotated at 2000 r.p.m.

Plate VIII

RTV-11 APPLICATION RESULTS

Trial No. 4--The same application procedure was used as in trial No. 1, except that Polylease-77 was used for the release agent.

It was noted that, as in all previous dipped trials, the application of the catalyst was accompanied by an increase in the dripping of the compound. This appears to be due to the additional weight of the catalyst or to a decrease in viscosity caused by the catalyst. Also noted was the folding of the membrane surface, which had begun to roughen the surface as in test No. 1.

After a ten hour time lapse, during which the membrane was unobserved, the majority of the RTV had fallen to the floor, leaving a thin film of uncatalyzed RTV on the mandrel.

Trial No. 5--A mandrel was presprayed with Silicure T-773, dipped into the uncatalyzed RTV-11 compound, and briefly allowed to drain. Then, before completely drained, it was rotated at 80 r.p.m. in order to retain a greater quantity of the compound on the mandrel. While rotating, the mandrel was sprayed with Silicure T-773 to catalyze the RTV.

Plate VIII-2 illustrates the surface texture obtained in this trial. The texture shown is similar to those of trials (1) and (4). The texture began to roughen when the membrane was sprayed with the catalyst. The roughness was magnified toward the tip of the mandrel due to eccentric rotation.

Since a greater amount of RTV had been retained on the mandrel by rotation, the cause of this and previous failures could be determined. The Silicure T-773 used for the release agent catalyzed a thin layer of the compound adjacent to the mandrel, and when sprayed on the dipped mandrel, it catalyzed a thin layer at the outer surface. This thin outer layer prevented any additional catalyst from penetrating into the uncatalyzed, viscous middle layer. Thus, as the middle layer flows down the mandrel, the outer surface of the membrane becomes more and more irregular. This downward movement will continue until the outer layers fall from the mandrel, as in trial (4), or until the folding of the outermost layer is great enough to penetrate through the middle layer to the other catalyzed layer and enough frictional resistance is produced to prevent the downward flow of the middle layer.

Trial No. 6--The RTV-11 was catalyzed with 1% Thermolite-12 and applied to a mandrel which had been presprayed with Polylease-77. During application, the mandrel was rotated with its axis horizontal at 80 r.p.m. so that the compound could be applied in a manner similar to that of a lathe. After application, the mandrel was rotated in a vertical position until the compound cured. In this vertical position the compound, being much less viscous than RTV-60, was able to flow into a uniform thickness.

A few small particles were noted in the membrane. It is believed that they were caused by one or a combination of the following:

1. The age of the RTV-11. (General Electric had suggested that the RTV be used before the time of this trial.)

2. Exposing the uncatalyzed RTV-11 compound to the air while the dipped mandrels were draining. (Each mandrel was drained for approximately one hour.)

3. Dipping and draining into the uncatalyzed RTV-11 compound a mandrel presprayed with Silicure T-773 Catalyst

Spray. (The spray was allowed to dry before dipping the mandrel.)

This type of application was deemed impractical, as the thickness of the membrane could not be controlled.

Trial No. 7--The same application procedure as in trial (6), but the rotational speed of the mandrel during application was 2000 r.p.m.

Application was found to be impossible, as the spinning mandrel seemed to repel the compound. Therefore, a complete coverage of RTV-11 was applied while the mandrel was at a standstill. When the mandrel was again rotated at 2000 r.p.m., most of the compound was thrown off by centrifugal force, leaving a very thin membrane of fairly uniform thickness on the mandrel. Plate VIII-3 shows the resulting membrane and the mandrel on which it was formed.

Application during high speed rotation seems to be impractical for catalyzed RTV compound due to the loss of the compound thrown off by centrifugal force. However, this type of application did seem appropriate for aerosol RTV, since the spray coatings might be thin enough and/or viscous enough to be unaffected by centrifugal force.

The application attempts at using the RTV-11 Silicone Rubber are summarized in Table VI.

Recommended Manufacturing Procedure

As previously stated, the procedure for manufacturing membranes should be uncomplicated and economical. Also, the procedure should be consistent--i.e., produce membranes of

TABLE VI

APPLICATION ATTEMPTS WITH RTV-11 SILICONE RUBBER

TRIAL NO.	1	2	3	4	5	6	7
RELEASE AGENT	Silicure T-773 Spray	Silicure T-773 Spray	Polylease- 77	Polylease- 77	Silicure T-773 Spray	Polylease- 77	Polylease- 77
CATALYST	Silicure T-773 Spray	Silicure T-773 Spray	2%Therm- olite-12	Silicure T-773 Spray	Silicure T-773 Spray	1%Therm- olite-12	1%Therm- olite-12
TYPE OF APPLICA- TION	Dipped in uncatalyzed compound and drained	Dipped in uncatalyzed compound and drained	Catalyzed compound, brushed or poured on	Dipped in uncatalyzed compound and drained	Dipped in uncatalyzed compound, then spun @ 80 r.p.m.	Brushed at 80 r.p.m.	Brushed- stationary application, then spun @ 2000 r.p.m.
SURFACE TEXTURE	Smooth prior to T-773, rough and folded after	Evenly dis- tributed, but tacky never cured	Smooth with few runs	Fell off, tacky film remaining never cured	Rough and irregular	Smooth and undulating	Smooth with large "orange- peel"
FINAL MEMBRANE THICK- NESS	Varied, pulled to tip	Could not be mea- sured	Too thick, approxi- mately 17- 18 mils	Thin	Varied, pulled to tip	About right, approxi- mately 7-8 mils	Too thin, approxi- mately 2 mils

uniform thickness and texture. Hence, the spray procedure is recommended as the method of application best suited for the purpose. Moreover, the rotating spray application is preferred over the stationary spray application, as the former seems to lessen the individual technique required to eliminate the "orange-peel" texture.

The brush application is deemed undesirable due to the nonuniform membranes obtained with this procedure. The irregularities incurred by this method might be eliminated if the material were viscous enough to flow into the desired texture and uniform thickness. This, however, would be comparable to either the pouring or the dipping type of application.

The dipping of mandrels into the uncatalyzed compound, then applying the catalyst, is not advised due to the previously described results. Dipping into a catalyzed compound might prove to be satisfactory, except that the time required to drain the surplus material from the mandrel utilizes the majority of the working time. Thus, unless several mandrels can be dipped and drained simultaneously (before expiration of the working time), a considerable wastage of material will be incurred.

CHAPTER IV

LABORATORY TESTS

Triaxial Swelling Test

This test was performed during the developmental stage of the testing apparatus. Thus, the primary purpose of the test was to observe the apparatus during operation and to see if it functioned as planned. The procedure used for apparatus assembly and testing was similar to the procedure presented in Chapter II. The data obtained from this test is given in Table VII, and Figure 9 shows the swelling-time curve for the specimen tested. The test data are presented in order to indicate the degree of accuracy which may be obtained with this apparatus. With practice, the individual will become adept in the operational procedure and small discrepancies in the test data can be eliminated.

During the test, the performance of the apparatus left little to be desired. No leakage could be detected at the O-ring seals or at any of the connections. All visible air bubbles were readily eliminated before starting the test.

As previously mentioned, the only malfunction was the effect of the silicone oil chamber fluid on the latex membrane.

Date	Temp (°C)	Time	∆ Time (min)	Dial Rdg. (in)	∆h _s (cm)	$\Delta v_{s}^{(\Delta h)}$ (cc)	E (%)	W.L. (cm)	ΔW.L. (cm)	V _W (cc)	L _t (cm)	ΔL_t (cm)	$\Delta v_{s}^{(\Delta d)}$	∆d _s (cm)	٤. (%)	Ey Ex
6-15	22.9	1648	0	1.0000				4.2247			13.82					
		1649	1	1.0011	.0028	0.0278	0.0394	4.3828	.1581	1.302	17.30	3.48	0.161	.005	0.1132	0.348
		.1650	2	1.0022	.0056	0.0556	0.0787	4.3810	.1563	1.288	19.20	5.38	0.249	.006	0.1730	0.455
		1652	4	1.0038	.0097	0.0963	0.1364	4.3737	.1490	1.227	20.50	6.68	0.309	.008	0.2172	0.628
		1710	22	1.0107	.0272	0.2702	0.3825	4.4181	.1934	1.593	25.43	11.61	0.537	.013	0.3772	1.014
	۰.	1750	62	1.0198	.0503	0.4996	0.7073	4.4713	.2466	2.031	29.41	15.59	0.721	.018	0.5067	1.396
6-16	.21.2	0825	937	1.0606	.1539	1.5286	2.1639	4.7352	.5105	4.205	42.95	29.13	1.348	. 033	0.9437	2.293
	21.9	1021	1053	1.0622	.1580	1.5693	2.2216	4.7352	. 5105	4.205	43.55	29.73	1.375	.034	0.9631	2.307
6-19	21.3	1015	5367	1.0760	.1930	1.9170	2.7137	4.8844	. 6597	5.434	48.12	34.30	1.587	. 039	1.1103	2.444
	21.6	1719	5791	1.0761	. 1933	1.9199	2.7179	4.8844	.6597	5.434	49.12	35.30	1.633	.041	1.1425	2.379
6-22	21.3	0809	9561	1.0791	.2009	1.9954	2.8248	4.9555	.7308	6.020	49.12	35.30	1.633	.041	1.1425	2.472
·	Where:	$ \begin{array}{c} \Delta h_{s} \\ \Delta v_{s}^{(\Delta h} \end{array} $	= chang) = chang	ge in len ge in volu	gth of thume of th	he sample he sample	due to ∠	h _s	$L_t = \Delta L_t $	position relative	n of the e moveme	interfa nt of th	ce in the e interfa	saran .ce	tubing	

TABLE VII TRIAXIAL SWELLING TEST DATA

∆ds = change in diameter of the specimen

- $\Delta W.L. = change in W.L.$
- εχ = lateral strain

 V_w = volume of water which has entered the sample

W.L. = level of water in the reservoir



Figure 9

Triaxial Swelling Ratio-Time Curve

During the latter part of the test, the membrane was observed to be hanging loosely around the specimen. Upon disassembly of the apparatus, the degenerated condition of the membrane was noted. The rubber was completely inelastic and fell apart when handled. The thickness of the membrane had decreased from 0.0043 inches before the test to 0.0026 inches after the test. Moreover, the weight of the membrane had increased from 2.1458 grams before the test of 4.3601 grams after the test.

At the conclusion of this test, it was felt that an adequate testing apparatus had been developed. However, since "a chain is only as strong as its weakest link," the investigative efforts were directed toward the development of a more satisfactory membrane, rather than the continuance of the swelling tests.

For this test, the following numerical values were used in the appropriate calculations:

Water Reservoir:	I.D.	=	1.2750	in	=	3.2385 cm	=	$\mathbf{d}_{\mathbf{W}}$
	Area	=	1.2768	in^2	H	8.2377 cm^2	=	Aw
Soil Specimen:	Diam.	-	1.40 in		=	3.556 cm	=	ds
	Height		2.80 in		-	7.112 cm	=	hs
	Area	-	1.5394	in^2	=	9.9321 cm^2	=	As
Saran Tubing:	I.D.		0.0956	in	=	0.2428 cm	=	dt
	Area		0.00717	in^2	=	0.04626 cm^2	-	At

In order to simplify the calculations, the following assumptions were made:

1. The diameter of the specimen remains constant for

vertical expansion calculations.

2. The height of the specimen remains constant for lateral expansion studies.

3. Lateral swelling of the specimen may be represented by an average increase in the diameter.

Thus, the lateral strain may be directly obtained as follows:

Volume change = V'_S - V_S , which is measured in the saran tube.

By substitution and solving for the final diameter,

$$d'_{s} = \sqrt{\frac{(\Delta L_{t})(d_{t})^{2}}{h_{s}} + (d_{s})^{2}} = d_{s} \sqrt{\frac{(\Delta L_{t})(d_{t})^{2}}{h_{s}(d_{s})^{2}} + 1}$$

where V'_{S} = the expanded volume of the specimen V_{S} = the original volume of the specimen d'_{S} = the expanded diameter of the specimen ΔL_{t} = the relative movement of the interface in the saran tubing.

Thus, with assumption (2), the lateral strain becomes:

$$\xi_{x} = \frac{d_{s} - d_{s}}{d_{s}} = \sqrt{\frac{(\Delta L_{t})(d_{t})^{2}}{h_{s}(d_{s})^{2}} + 1} - 1$$

Substituting the numerical values for this test into the above equation,

$$\mathcal{E}_{x} = \sqrt{\frac{(0.2428)^{2}}{7.112(3.556)^{2}}} (\Delta L_{t}) + 1 - 1$$

$$= \sqrt{0.000656 \Delta L_{t} + 1} - 1 , \text{ in which } \Delta L_{t} \text{ is ex-pressed in cm.}$$

Tests on Membranes

As it happened, the development of a reliable procedure for the fabrication of silicone rubber membranes occupied a major portion of the time available for the entire investigation. Therefore, comparatively little time was devoted to the testing of the membranes. The tests presented here are not to be viewed as final quantitative studies, but rather as guides for future comprehensive investigations.

Osmosis Test

The pore water of many natural clays possesses a certain salinity. When distilled water, or water of low mineral content, is used to supply the pressure in the confining chamber during a triaxial test, the difference in salinity of the water on the two sides of the membrane used to enclose the soil specimen is responsible for the development of osmotic pressures across the membrane. These pressures result in a flow of water through a semi-permeable membrane in such a way as to dilute the more concentrated solution. As a consequence, in tests of long duration, there is a tendency for the moisture content of the soil specimen to in-Such variations in moisture content have direct, crease. though unpredictable, effects on the test results. Obviously, a membrane which reduces the osmotic flow to a minimum is highly to be desired.

For clarity, the following description (from reference 8) of the process of osmosis is proffered:

OSMOSIS - If a membrane into which a solvent readily penetrates is used to separate two solutions of different concentrations, we invariably find that some of the solvent passes through the membrane from the less concentrated into the more concentrated solution. The level of the more concentrated solution will therefore slowly rise.

The incoming solvent, penetrating through a membrane from a pure solvent into a solution, sets up a pressure called the OSMOTIC PRESSURE. We can measure this by noting what pressure needs to be applied to the solution to stop the entrance of solvent through the membrane. The osmotic pressure may amount to many atmospheres, in solutions of moderate concentration.

A membrane through which a solvent penetrates, while the solute is held back, is called a SEMI-PERMEABLE MEMBRANE. The flow of the solvent through a membrane, from a less concentrated into a more concentrated solution, is called OS-MOSIS, (or occasionally, OSMOTIC FLOW).

Osmosis need not seem mysterious. A dissolved solute decreases the tendency of a solvent to escape from a solution into a membrane for the same reasons that it reduces the tendency of the solvent to escape into a vapor. So, if we have a membrane wet by solutions of two different concentrations, molecules from the low-concentration solution penetrate that side of the membrane more frequently, and return from the membrane less frequently, than they do on the high-concentration side. The result is a continuous flow of solvent through the membrane.

To investigate the efficiency of a silicone rubber membrane in reducing osmotic flow, an RTV-60 membrane was subjected to conditions as severe as those which are ever likely to be encountered during actual soil testing. Specifically, a three per cent solution of salt (the approximate equivalent of sea water) was placed on one side of the membrane and distilled water on the other.

After filling the RTV-60 membrane with the salt solution, a rubber stopper was inserted and the membrane tightly bound over it with a rubber strip. The sealed membrane was then immersed in distilled water, as shown in Plate IX-1. Over a four month period, the volume and weight of the sealed



1. Osmosis Test



2. Extension Modulus Test

Plate IX TESTS ON MEMBRANES

Date		e	Days	Volume* (cc)	Weight** (gms.)	
13	Nov	61	0	211	223.19	
27	Nov	61	14	211	225.29	
11	Dec	61	28	215	227.90	
16	Jan	62	64	218	241.10	
28	Mar	62	135	225	238.50	

membrane were checked and recorded as follows:

*A 500 ml. graduated cylinder was used to measure the amount of water displaced.

**Submerged in a 1000 ml. beaker (the difference between the weight and volume is due to the average specific gravity of the materials being greater than that of water).

Upon completion of the test, the individual items used in the test were weighed, and the following results obtained:

	Dry	Wet	Dry		
	(before test)	(after test)	(after test)		
Membrane	6.4606 gms.	6.3500 gms.	6.3263 gms.		
Rubber Stopper and Strips		43.9885 gms.	42.9232 gms.		

Salt	Solution	plus	Tare	357.57	gms.
S	Salt	plus	Tare	176.69	-
		Water		180.88	
			Tare	171.64	
			Salt	5.05	

The unexpected decrease in the weight of the membrane indicates that perhaps a portion of the membrane went into solution in either the salt solution or the distilled water. This also might possibly explain the slimy appearance of the inside surface of the membrane and the fungus-like particles in the salt solution which were noted at the conclusion of the test. An indication of the osmosis which occurred may be obtained by comparing the concentration of the salt solution inside the membrane before and after the test. Assuming that a correct weight for the solution inside the membrane was found after the test and that the amount of salt within the membrane did not change, the amount of water which entered the membrane by osmotic flow is approximately:

$$180.88 - \frac{5.05}{0.03} = 180.88 - 168.33 = 12.55 \text{ cc.}$$

The quantity of water passing through the membrane is also indicated by the change of volume (14 cc.) of the membrane and its contents during the test. However, a portion of this change may perhaps be ascribed to swelling of the membrane, stopper, etc.

Osmosis studies conducted by R. E. Moreland (9) have indicated that, for latex membranes, the osmotic flow rate is probably not in excess of 0.0015 cc. of water per cm² of membrane area per day. The surface area of the RTV-60 membrane used in this test was approximately 200 cm². For a latex membrane with this same surface area, the amount of water which would have entered the membrane would "probably not be in excess" of 40 cc. (.0015 x 200 x 135).

It would appear from the comparison of the two osmotic volumes that the silicone rubber is much more effective in the prevention of osmosis. Even though the data above are quite skimpy, it may be inferred that a thinner silicone rubber membrane may be used to achieve the same results obtained with the standard latex membrane.

Water Absorption Tests

Various samples of RTV rubber were immersed in distilled water in an attempt to determine the change in weight due to water absorption which might be expected for any given membrane. These tests were also performed with the thought in mind that the absorptive capacities of membrane materials might be related to, or indicative of, the osmosis which occurs through membranes. The results of the tests are as follows:

(1) RTV-60 membranes (approximately 6 mils thick) used in triaxial shear strength tests.

Test	Duration	Change	in	Weight
1	week	+	0.0	092%
2	weeks	+	1.3	333%

(2) Various thicknesses of RTV-11 and RTV-60 samples, immersed for two weeks in distilled water.

	Approximate		Change in	Weight (%)		
	Thickness	Unstre	tched*	Stretched*		
Material	(mils)	Wet**	Dry**	Wet**	Dry**	
RTV-11	2	- 0.31	- 0.84	+ 3.6	- 1.53	
	7	+ 0.062	- 0.60	- 0.22	- 0.88	
	16	+ 0.25	- 0.04	+ 0.405	0.0	
RTV-60	2	+ 0.33	- 3.67	+ 3.38	- 1.29	
	6	+ 2.44	- 0.78	+ 3.28	- 0.88	

*In the extension modulus tests (explained later), an initial elongation of the membrane seemed to alter the elastic properties of the membrane. It was thought that it might make some difference in the amount of water absorbed.

**The sample was weighed prior to immersion, and this weight compared with the weight obtained immediately after removal from the water. The initial weight was also compared to the weight obtained after the sample had dried for approximately 24 hours. (3) Various membranes of RTV-11 and RTV-60, immersed for $5\frac{1}{2}$ days in distilled water.

		Change in	Weight (%)		
Membrane		Wet	Dry		
RTV-11	(17 mils)	- 0.0275	- 0.0841		
RTV-11	(2 mils)	- 0.0838	- 0.0915		
RTV-60	(2 mils)		- 1.816		
RTV-60*	(sprayed				
	on plate)	- 0.443	- 0.516		
RTV-60*	(standstill)	- 0.283	- 0.476		
RTV-60*	(400 r.p.m.)	- 0.413	- 0.600		
RTV-60*	(600 r.p.m.)	- 0.362	- 0.473		

*Average thickness of 6 mils.

The results of the above tests were, to say the least, surprising and totally unexpected. The published data from General Electric gives the resistance to immersion in numerous fluids (not distilled water, however), and in no instance indicates a decrease in weight. It should be noted that the results of this investigation show that in all but one instance (zero change in the thick RTV-11 sample), a decrease in weight was experienced after immersion in distilled water. This phenomenon may perhaps be inherent to silicone rubber, but a definite reason for this behavior is unknown at this time.

Extension Modulus Tests

These tests were performed in order to obtain another comparison between latex and silicone rubber membranes. Henkel and Gilbert (10) made load-extension tests, as shown in Figure 10, in order to determine the elastic properties of thick latex membranes (approximately 20 mils thick), standard commercial latex membranes (approximately 8 mils thick), and thin membranes (approximately 4 mils thick).



Figure 10

Load-Extension Tests by Henkel and Gilbert

Their results for the three types of membranes are shown in Figure 11. They state:

Although the load/strain relation is slightly curved, the different rubber (latex) membranes have been characterized by an average extension modulus defined as load per inch for unit strain. For the thick, standard, and thin rubber membranes the extension moduli are 3.4, 1.5, and 0.8 lb. per inch respectively.

Upon checking the graph of their results, it appears that the extension moduli are obtained by using a ten per cent secant modulus for each membrane.



Load/Strain Curves for Three (Latex) Rubber Membranes

The load-extension tests for this investigation were performed as shown in Plate IX-2. The mean length of the membrane was measured directly by pressing it flat and measuring with a scale which was graduated in 0.01 inch increments. The scale was read to the nearest 0.001 of an inch with the use of a lOx magnifying glass. The weight of the scale pan was considered sufficient to take up all slack in the membrane prior to the addition of loads, but was neglected in calculating the load applied to the membrane. Its weight was approximately 24 grams. The approximate thicknesses of the membranes which were tested are shown below.

	Approximate	Membrane	Thickness	
	Thick	Medium	Thin	
RTV-11	17 mils	7 mils	2 mils	
RTV-60		6 mils	3 mils	

Three different load cycles were used for this investigation: a rapid loading (R), in which the loads were added as quickly as possible; a creep loading (C), in which the loads were applied at regular time intervals and the elongation measured at the beginning and end of each interval; and a load-unload cycle (L), for which the elongation was measured during unloading as well as loading. Figures 12 through 22 show the results of various load cycles for each membrane tested. "Typical values" for the various thicknesses of each material are shown in Figures 23, 24, and 25. Using a ten per cent secant modulus (as was done for latex), the average extension moduli for the different silicone rubber membranes are as follows:

	Approximate Extension Moduli (lb/inch)		
	Thick	Medium	Thin
RTV-11	6.4	2.2	0.7
RTV-60		1.8	0.9

It was noted in the testing of each membrane that the elongation during the initial load cycle was much less than during subsequent loadings--i.e., for each membrane tested, the initial load/strain relation is unique. This characteristic can be seen in the graphs of the results (Figures 12-22). Also, the load/strain relations for all loading cycles, which succeed the initial cycle, appear to fall into a uniform pattern characterized by a decrease in the extension modulus,
particularly in the 30-50% strain region. Membranes which were retested after a period of time (as much as a month) showed an increased extension modulus, but the load/strain curve was still within the pattern mentioned above.

During the initial loading cycle, after attaining approximately twenty per cent strain, the deflection of every membrane under each additional load followed the general pattern shown below.



Upon the addition of a load, the membrane immediately deflects from (1) to (1), then very rapidly to (2), and after some time to (3). If the load is removed, the membrane will quickly return to (4), then gradually to (5), leaving a small amount of elongation ((0) - (5)) "built-in" the membrane. For all subsequent loading cycles, the addition of the same load increment will cause the membrane to immediately deflect from (5) to (2), and with time to (3). Upon removal of the load, the membrane will return to (5), through (4), as in the initial loading.

Throughout the initial loading cycle, the "built-in" elongations for each load increment (after 20% strain) are apparently compounded and collectively produce the decrease in the extension modulus previously mentioned. The deflections for three successive rapid loading cycles (Sample 2, RTV-60-Medium) shown below illustrate the effect of the initial loading upon the subsequent loadings.

	Total	Deflection	(inches)
Load (gms)	Initial Loading	Second Loading	Third Loading
0	0	0	0
20	0.010	0.011	0.012
40	0.029	0.030	0.031
60	0.045	0.048	0.055
80	0.065	0.068	0.080
100	0.085	0.090	0.109
150	0.140	0.150	0.170
200	0.196	0.210	0.241
400	0.392	0.510	0.550
600	0.570	0.780	0.830
800	0.730	0.970	1.000
1000	0.880	1.080	1.121
1200	1.010	1.105	1.202
1500	1.190	1.240	1.290
0	0.010	0.000	0.000

The elastic properties of the membrane do not appear to be appreciably affected by the initial loading cycle until approximately twenty per cent strain is induced. This amount of strain probably exceeds the maximum which will be incurred in either the triaxial shear strength test or (especially) the proposed swelling tests (only 1.14% lateral strain occurred in the swelling test previously described in this chapter). Thus, it is believed that, under normal laboratory conditions, the quasi-elastic deformation and "built-in" elongation will not occur in the magnitudes indicated in this investigation.



Load-Strain Curve for RTV-11 (#1 - Thin)





Load-Strain Curve for RTV-11 (#2 - Thin)





Load-Strain Curve for RTV-11 (#1 - Medium)



Load-Strain Curve for RTV-11 (#2 - Medium)



Figure 16

Load-Strain Curve for RTV-11 (#1 - Thick)



Figure 17

Load-Strain Curve for RTV-11 (#2 - Thick)





Load-Strain Curve for RTV-60 (#1 - Thin)



Figure 19

Load-Strain Curve for RTV-60 (#2 - Thin)



Figure 20

Load-Strain Curve for RTV-60 (#1 - Medium)





Load-Strain Curve for RTV-60 (#2 Medium)



Figure 22

Load-Strain Curve for RTV-60 (#3 Medium)



Average Load-Strain Curve for RTV-11 and RTV-60 (Thin)



Average Load-Strain Curve for RTV-11 and RTV-60 (Medium)



Average Load-Strain Curve for RTV-11 (Thick)

CHAPTER V

CONCLUSIONS AND RECOMMENDATIONS FOR FUTURE RESEARCH

Swelling Test Apparatus

The swelling test apparatus was designed and developed in order to obviate some of the unfavorable characteristics inherent in currently used testing devices.

The test accomplished in the initial stage of development indicates that the apparatus evolved in this investigation performs quite adequately. However, as the apparatus was subjected to only one test, no final conclusions pertaining to the quality of performance of the apparatus are formulated at this time.

It is believed that final evaluation of the performance of the apparatus can be accomplished only after it has been used for a while in conducting additional swelling tests.

Manufacture of Membranes

The membrane development investigations were occasioned by an unexpected malfunction of one of the commonly used latex membranes, and by the prospects of advantages to be

gained from the use of a less permeable membrane (particularly for long-term tests). On the basis of experience gained from the examination and testing of several RTV membranes, formed using various application techniques and procedures, it is believed that the method of spray application is to be preferred. This manufacturing procedure is favored on the basis of economy, ease of application, and control of membrane thickness. Moreover, it lends itself quite well to any size of laboratory operation--i.e., membranes can be manufactured singly or in groups of any number.

It has been suggested by Mr. Richard C. Scott of the Silicone Products Department of General Electric that future manufacturing attempts be extended to include a pour or brush coat of RTV-11 containing 0.3 per cent Thermolite-12 and 10 to 15 per cent of RTV-910 diluent oil. It was also suggested that perhaps a catalyst could be applied without solvent by flow coat to effect a smoother outer surface of the membrane. If the compound retained on the mandrel still appears to be too thick, an air hose might serve to blow off the excess compound, thus reducing the membrane thickness.

Properties of Membranes

In order to obtain a more comprehensive comparison between the standard latex membrane and the proposed silicone rubber membrane, it was desirable to carry out various tests of the properties of silicone rubber membranes. The results of the tests could then be compared, at least qualitatively, with similar data pertaining to latex.

The osmosis test accomplished during this investigation yielded favorable results in terms of the reduction of osmotic flow through the membrane. The test indicated that the use of membranes of silicone rubber might reduce the osmotic flow as much as 60 per cent, compared to latex membranes of the same thickness.

Future membrane investigations should be primarily concerned with osmotic studies, as the passage of excessive osmotic flow constitutes one of the principal defects of latex membranes.

The water absorption tests which were conducted indicate that from this standpoint the silicone rubber is superior to latex, which during previous usage has been found to absorb about 10 per cent water, by weight. This compares with about a 1 per cent increase in weight noted for **RTV-60** membranes used under similar conditions.

It is believed that the results obtained in this investigation should be verified by continued testing before conclusive statements are drawn, since it appears that some slight dissolution of the material may occur in distilled water.

The elastic properties of the silicone rubber membranes were thoroughly investigated by means of rapid loading and creep loading tests. On the basis of the test results, it is concluded that the extension modulus is only slightly greater than that of natural rubber latex. The load stress-strain characteristics of the silicone rubber are believed to be at least as desirable as those of natural rubber latex. For low strains (below about 20 per cent), the behavior seems to be nearly elastic. For larger strains a quasi-elastic effect, followed by creep deformation results, with time, from each increment of load. As a result, some permanent set occurs during the first loading cycle. It is not known whether repetitious loads involving only strains of less than 20 per cent will eventually result in some permanent set. The answer to this question should be obtained from further research. However, it is believed that the small permanent set which does occur is not objectionable. In fact, it may in certain respects be desirable, since it implies a decrease in the confinement offered by the membrane during tests in which a slow lateral spreading of the specimen follows the application of axial loads or the absorption of water (during swelling tests).

It is finally concluded that the silicone rubber membranes appear to be generally superior to latex membranes for the performance of (especially) long-term tests. Routine use of the membrane under normal laboratory conditions will eventually permit a final evaluation to be made.

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