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Long-Term Hydrostatic Tests of Syntactic Foam

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CONTENTS

Abstract	iv
Problem Status	iv
Authorization	iv
INTRODUCTION	1
GENERAL	1
TEST OBJECTIVES AND PROCEDURES	3
TEST DATA EXPLANATION	3
DISCUSSION	23
CONCLUSIONS	24
APPENDIX – The Use of Sonic Energy for the Measurement of Some Physical Properties of Syntactic Foam	32
ACKNOWLEDGMENT	34

ABSTRACT

Syntactic foam samples were tested in fresh water under pressures of 3000, 5000, and 10,000 psi at 2°C and at room temperature (about 25°C). The samples were 1-3/4-in.-diam, 6-in.-long solid cylinders. The long-term hydrostatic test results, presented here, indicate that the rate of water absorption continues almost linearly with exposure time.

Based on the data presented at the present time the life expectancy (no longer buoyant) of a good grade of 42-lb/ft^3 syntactic foam at 10,000 psig is estimated to be about 10 years.

PROBLEM STATUS

This is a complete report on one phase of the long-term hydrostatic testing of syntactic foam.

AUTHORIZATION

NRL Problem S01-20
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LONG-TERM HYDROSTATIC TESTS OF SYNTACTIC FOAM

INTRODUCTION

The Naval Research Laboratory was assigned the task of building and installing a submerged oceanographic platform for the Atlantic Underwater Test and Evaluation Center (AUTEC). This platform is to be in the form of a winch – located at the middepth level of the ocean. The platform is expected to operate satisfactorily for a minimum, unattended period of one year. To buoyantly support such a winch system and its attendant mooring cables requires a large amount of floatation material. Due to the variety of float material available, an investigation of the most promising float systems was undertaken.

GENERAL

The first float material considered was glass. Hollow glass spheres, of the fishnet-float type, are relatively inexpensive and are capable of providing a high-excess positive buoyancy-to-weight ratio at hydrostatic pressures up to 10,000 psi. After performing various hydrostatic pressure tests on 4-in.-diam hollow glass spheres, the investigation was discontinued. Although the glass spheres had most of the desirable qualities for a high-pressure floatation system, it was found that a failure in one sphere among a group of spheres sympathetically imploded all the other glass spheres in its vicinity. For this reason, no further evaluation of glass floats was pursued.

Positive buoyancy is obtainable by use of metallic or fiberglass pressure vessels and by use of low-density liquid or solid systems. Sufficient data are available on most of these types of float systems; so they were not investigated. Table 1 gives some pertinent physical and mechanical properties of materials used in float systems.

One float system material which was just entering into the competitive market, for which little information was available, showed promise of meeting our requirements. This material, referred to as syntactic foam, uses a low-density filler material suspended in a matrix of epoxy resin. The fillers used in syntactic foam are microscopic-sized hollow glass or phenolic spheres. This report deals with the test procedures and results of our investigation of this new type of float material.

The types of tests to which the float material was subjected were not of a general nature but were directed specifically toward the requirements of the submerged winch system. In this application the float material would be deeply immersed into the ocean for about one year; it would then be stored ashore for three months and again immersed. The dominant physical test requirement for this application is the long-term exposure of the float material to hydrostatic pressure. Consequently, the dominant test condition investigated was long-term exposure to simulated environmental conditions of pressure and temperature.

It may be well to further describe this buoyant material. At present, this material is generally referred to as syntactic foam. It is also referred to as plastic foams, epoxy floats, glass bead floats and various other quasi-descriptive terms. The filler materials which are used are referred to as glass spheres, microspheres, eccospheres, glass beads, glass balloons and microballoons. Similar hollow spheres are also made of a

Table 1
Properties of Buoyancy Materials for Underwater Systems
with All Values Given for 20°C

Test Material	Compressive Strength S_c (psi)	Density ρ at 1 Atm. (lb/ft^3)	Young's Modulus E (psi $\times 10^{-6}$)	Bulk Modulus K (psi $\times 10^{-6}$)	Poisson's Ratio μ	Relative Compressibility β, $\Delta P = 10{,}000$ (%)	Acoustic Impedance ρc (lb/ft^2-sec $\times 10^{-5}$)	Excess Buoyancy B_0 (lb)*	Excess Buoyancy B_1 (lb)†	Excess Buoyancy B_{12} (lb)‡
Syntactic Foam	17,000	42.0	0.42	0.35	0.30	1.8	2.60	22.0	23.0	--
Steel	150,000	490.0	30.0	24.6	0.30	0.0413	80.0	-3.1	-1.5	28.4
Aluminum	60,000	168.0	10.8	10.6	0.33	0.0943	29.0	8.1	9.7	34.2
Titanium	120,000	282.0	16.9	15.5	0.32	0.07	47.0	16.0	17.6	37.5
Lithium	--	33.3	1.66	1.61	0.426	0.58	3.34	30.7	32.3	--
Glass	>300,000	150.0	8.9	4.6	0.17	0.212	29.2	52.4	53.9	--
Ceramic	>300,000	120.0	50.0	4.62	0.32	0.0216	74.2	47.6	48.4	--
Polyethylene	3,500	59.0	0.015	0.362	0.458	2.8	3.28	5.0	5.1	--
Sea Water	--	64.0§	--	0.345	--	2.8	3.19	0	0	--
Fresh Water	--	62.4	--	0.309	--	2.7	3.03	1.6	1.7	--
Gasoline	--	44.0	--	0.146	--	5.6	3.75	20.0	19.3	--

*B_0 = external pressure = 1 psig.
†B_1 = external pressure = 10,000 psig.
‡B_{12} = external pressure = 10,000 psig with 5000 psig internal pressure (Helium-filled, wt. of He at 5000 psig = 4.05 lb/ft^3).
§Sea water at 20°C and 10,000 psig = 65.79 lb/ft^3.

plastic material. Only the agglutinating system seems to have one generally accepted appellation -- namely, epoxy resin.

Syntactic foam is neither a buoyant pressure vessel nor a pressure-compensated container. It may be described as a mechanical mixture of a large number of small pressure vessels held in solid suspension by a resin. The filler materials, or hollow spheres, provide the positive buoyancy — the resin holds the spheres together.

Of novel interest are the hollow glass beads, or microballoons. This product has been produced for use as light reflectors on motion picture screens, paints for road signs, etc. In these applications the strength and quality of the glass bead used is of little consequence. For the glass-bead application in high-pressure floats, the quality and strength of the glass bead determine, to a considerable degree, the strength of the finished product. These glass beads, which in bulk form look like a white powder, have diameters ranging from 30 μ to 300 μ. The wall thickness of the beads is approximately 2 μ. A silica-grade or a sodium-grade glass is normally used in the manufacture of the glass beads. The phenolic grade of balloons are most often made from a urea-formaldehyde plastic.

The matrix in which the glass spheres are embedded is a high-compressive-strength epoxy resin. The percentage of filler material (glass beads) to resin has a very marked effect on buoyancy as well as on the compressive strength of the final product.

The density of syntactic foam is available from 20 lb/ft^3 to 50 lb/ft^3. The greater the percentage of glass beads to resin, the lower is the density of the float material. However, the lower density product results in a float having less compressive strength. Syntactic foam is made by a casting process; hence, almost any shape may be obtained by use of a suitable mold.

TEST OBJECTIVES AND PROCEDURES

To determine the usefulness of the syntactic foam as a buoyancy material, measurements were made to obtain (a) the effects of temperature and pressure under long-term hydrostatic exposure, (b) the effect of storage at atmospheric conditions after having been exposed to hydrostatic pressure, and (c) the effect of an overcoating of urethane on water absorption as compared to uncoated samples.

The answers to the above objectives were hoped to be extracted from an analysis of (a) the change in weight of a sample due to water absorption, (b) the change in volume of a sample due to water absorption and structural deformation, and (c) the change in buoyancy of the sample as measured under atmospheric conditions.

This information was obtained from physical measurements of the samples every two months. After two months at 10,000 psig, the samples were removed from the pressure bottles, dried, weighed, and its dimensions accurately determined. Buoyancy was measured directly by a water-displacement method. Six measurements of the diameter and five measurements of the length of the sample were made. Weight measurements were made on a balance scale capable of providing a resolution of 1/10 g. Density measurements of the weight and volume of the sample were checked against the density measurement obtained by the water-displacement principle.

TEST DATA EXPLANATION

The samples of syntactic foam used for the tests at NRL were obtained from and tested in cooperation with two principal suppliers of this material. The physical size of

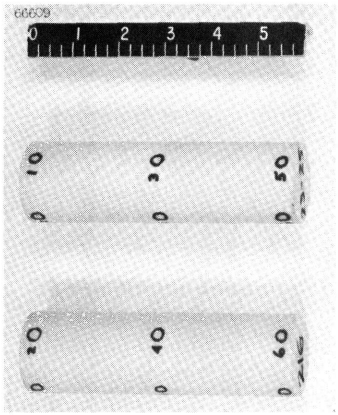

Fig. 1 - Uncoated syntactic foam samples

the test specimen for our tests was set by the 2-in. internal diameter of available pressure vessels. Two 1-3/4-in.-O.D., 6-in.-long specimens could be accommodated in one of these pressure vessels. Figure 1 shows some of the float samples used for these tests. Figure 2 shows a typical pressure bottle which was used for the long-term hydrostatic pressure tests. Fresh tap water was used as the pressurizing fluid for all tests.

Table 2 lists the test sample composition and pertinent physical data. Ultrasonic methods were used for the measurement of some of these physical properties. Two such methods are described in Appendix A.

Of the first five float material samples being tested, three samples were maintained at a pressure of 10,000 psig and a temperature of 2°C. The other two samples were subjected to 3000 psig and 2°C. One sample, designed for 3000-psi service, was made with a filler of phenolic beads, the other four were of the glass microballoon variety. Also, two of the samples were coated with 1/16-in.-thick polyurethane -- the remaining samples were uncoated. Test specimens were removed from the pressure bottle for measurements at two-month intervals.

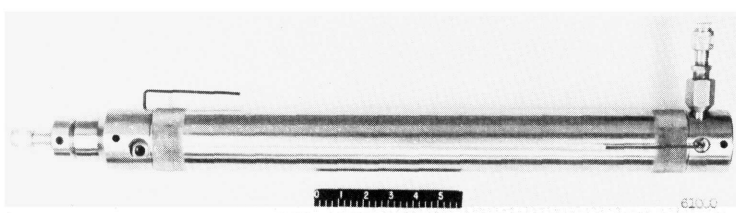

Fig. 2 - Hydrostatic test pressure bottle

Tables 3 present data on the first set of samples which were placed under test in December 1962. After being under hydrostatic pressure for one year, the samples were allowed to air dry at room temperature (about 25°C) and pressure for periods up to one year and were again immersed to their original pressurized state.

A second set of uncoated glass-bead type floats, of improved material, were placed under hydrostatic pressure of 10,000 psig about one year after the first samples. Two samples were stored at 25°C and two were stored at 2°C. The data being generated showed that a more stringent test condition existed for the specimen when stored at room temperature than at 2°C. From this point on, all samples were maintained at room temperature.

Tables 4 present test data on the second set of four samples to be placed under test. These samples are all uncoated. One sample of each composition was maintained at the two test temperatures for one year. After one year the test continued at room temperature.

In March 1966 four additional samples were placed under test. Two of these (density 38 lb/ft^3) are being subjected to 5000 psig; the other two (density 42 lb/ft^3) are being subjected to 10,000 psig. All four are being tested at room temperature.

Table 2
Composition and Physical Properties of Test Samples

Sample No.	Composition (Beads in Epoxy)	Coating	Dimensions		Volume (in.^{3})	Weight (lb)	Density (lb/ft^3)
			Diameter (in.)	Length (in.)			
1*	Phenolic	Urethane	1.865	5.843	16.02	0.395	45.2
2	Glass	Urethane	1.872	5.920	16.29	0.450	49.5
3	Glass	Uncoated	1.750	5.750	13.83	0.356	44.6
4	Glass	Uncoated	1.751	5.749	13.84	0.355	44.6
5	Glass	Uncoated	1.750	5.748	13.83	0.355	44.6
6†	Glass	Uncoated	1.752	5.752	13.82	0.363	45.0
7	Glass	Uncoated	1.750	5.753	13.80	0.358	45.0
8	Glass	Uncoated	1.744	5.764	13.74	0.360	44.8
9	Glass	Uncoated	1.748	5.757	13.81	0.358	44.8
10‡	Glass	Uncoated	1.764	5.994	14.62	0.323	37.7
11	Glass	Uncoated	1.760	5.997	14.55	0.317	37.7
12	Glass	Uncoated	1.749	5.998	14.35	0.356	42.4
13	Glass	Uncoated	1.745	5.992	14.27	0.352	42.4

*Set number 1.
† Set number 2.
‡ Set number 3.

Table 3a
Test Results for Sample 1 of the First Set of Samples

Date	Total Time (days)	Time Under Pressure (days)	Hydrostatic Pressure (psig)	Temp. (°C)	Medium	Density Increase (%)	Volume Decrease (%)	Water Absorption Percentage by Weight	
								Increase (%)	Avg. Increase (%/yr/10^4 psig)
12/14/62	0	0	3000	2	Water	--	--	--	--
2/8/63	56	56	3000	2	Water	1.17	0.42	0.73	15.8*
4/16/63	123	123	3000	2	Water	2.20	0.87	1.28	12.7
6/17/63	185	185	3000	2	Water	2.94	1.14	1.90	12.5
8/22/63	251	251	3000	2	Water	4.41	1.90	2.40	11.6
10/16/63	306	306	3000	2	Water	6.16	2.20	3.74	14.9
12/16/63	367	367	3000	2	Water	6.76	2.58	4.02	13.3
12/17/63	368	367	0	25	Air	--	--	--	--
1/21/64	403	367	0	25	Air	--	--	1.56	--
5/28/64	531	367	0	25	Air	--	--	1.45	--
7/16/64	580	367	0	25	Air	--	--	1.68	--
1/7/65	755	367	0	25	Air	--	--	1.73	--
2/10/65	789	367	0	25	Air	--	--	1.56	--
2/12/65	791	367	0	25	Air	--	--	1.57	--
2/15/65	794	367	0	25	Air	4.55	2.70	1.70	--
3/29/65	836	367	3000	25	Water	4.26	2.69	1.53	--

Table continues.

Table 3a (Continued)

Date	Total Time (days)	Time Under Pressure (days)	Hydrostatic Pressure (psig)	Temp. (°C)	Medium	Density Increase (%)	Volume Decrease (%)	Water Absorption Percentage by Weight	
								Increase (%)	Avg. Increase (%/yr/ 10^4 psig)
3/30/65	837	368	3000	25	Water	4.85	2.84	1.82	353.0†
3/31/65	838	369	3000	25	Water	5.00	2.97	1.84	189.0
4/1/65	839	370	3000	25	Water	5.00	2.88	1.92	158.0
4/2/65	840	371	3000	25	Water	5.00	2.84	1.99	140.0
4/5/65	843	374	3000	25	Water	5.44	3.08	2.14	106.0
4/6/65	844	375	3000	25	Water	5.44	3.12	2.19	100.0
4/7/65	845	376	3000	25	Water	5.58	3.14	2.30	104.0
4/8/65	846	377	3000	25	Water	5.73	3.24	2.29	92.5
4/9/65	847	378	3000	25	Water	5.88	3.25	2.41	97.4
4/12/65	850	381	3000	25	Water	6.17	3.49	2.48	82.6
4/13/65	851	382	3000	25	Water	6.32	3.48	2.52	80.3
4/14/65	852	383	3000	25	Water	6.46	3.56	2.58	79.8
4/15/65	853	384	3000	25	Water	6.61	3.72	2.66	80.9
4/16/65	854	385	3000	25	Water	6.76	3.86	2.64	75.1
4/19/65	857	388	3000	25	Water	7.05	3.94	2.74	70.1
4/20/65	858	389	3000	25	Water	7.05	3.97	2.79	69.7
4/21/65	859	390	3000	25	Water	7.20	3.95	2.81	67.8
4/22/65	860	391	3000	25	Water	7.34	4.16	2.83	65.9
4/23/65	861	392	3000	25	Water	7.49	4.17	2.99	71.1
4/26/65	864	395	3000	25	Water	7.93	4.54	3.01	64.3
4/27/65	865	396	3000	25	Water	7.79	4.39	3.04	63.4
4/28/65	866	397	3000	25	Water	7.93	4.47	3.08	62.8
4/29/65	867	398	3000	25	Water	8.08	4.53	3.11	62.0
4/30/65	868	399	3000	25	Water	8.08	4.60	3.11	60.1
5/3/65	871	402	3000	25	Water	8.52	4.85	3.22	58.7
5/4/65	872	403	3000	25	Water	8.52	4.78	3.28	59.1
5/5/65	873	404	3000	25	Water	8.52	4.80	3.39	61.2
5/6/65	874	405	3000	25	Water	8.96	5.06	3.39	59.6
5/10/65	878	409	3000	25	Water	9.69	5.58	3.52	57.7
5/12/65	880	411	3000	25	Water	10.14	5.84	3.63	58.1
5/17/65	885	416	3000	25	Water	10.43	5.98	3.73	54.6
5/20/65	888	419	3000	25	Water	10.43	5.82	3.93	56.2
5/24/65	892	423	3000	25	Water	11.02	6.28	3.99	53.4
5/28/65	896	427	3000	25	Water	11.32	6.45	4.08	51.8
6/1/65	900	431	3000	25	Water	11.60	6.62	4.21	50.9
6/4/65	903	434	3000	25	Water	11.90	6.80	4.28	50.0
6/10/65	909	440	3000	25	Water	12.34	6.92	4.44	48.5
6/16/65	915	446	3000	25	Water	12.92	7.33	4.66	48.2
6/21/65	920	451	3000	25	Water	13.07	7.31	4.76	46.8
8/3/65	963	494	3000	25	Water	14.85	7.87	5.78	40.7
2/14/66	1158	689	3000	25	Water	20.7	9.06	9.77	31.2
6/23/66	1287	818	3000	25	Water	26.2	11.0	12.1	28.5
8/24/66	1349	880	3000	25	Water	27.2	11.6	12.4	25.8
2/23/67	1532	1063	3000	25	Water	32.2	12.7	15.2	29.0

*0.73-0.00; 365/56; 10,000/3000.
†1.82-1.53; 365/1; 10,000/3000.

Table 3b
Test Results for Sample 2 of the First Set of Samples

Date	Total Time (days)	Time Under Pressure (days)	Hydrostatic Pressure (psig)	Temp. (°C)	Medium	Density Increase (%)	Volume Decrease (%)	Water Absorption Percentage by Weight	
								Increase (%)	Avg. Increase (%/yr/ 10^4 psig)
12/14/62	0	0	3,000	2	Water	—	—	—	—
2/8/63	56	56	3,000	2	Water	0.00	0.00	0.25	5.4
4/16/63	123	123	3,000	2	Water	0.39	-0.22	0.59	5.8
6/17/63	185	185	3,000	2	Water	0.52	0.00	0.54	3.6
8/22/63	251	251	3,000	2	Water	1.18	+0.19	0.98	4.8
10/16/63	306	306	3,000	2	Water	1.57	0.19	1.47	5.8
12/16/63	367	367	3,000	2	Water	2.09	0.60	1.47	4.9
12/17/63	368	367	0	25	Air	—	—	1.22	—
9/4/64	630	367	0	25	Air	0.78	0.26	0.49	—
9/4/64	630	367	10,000	2	Water	—	—	—	—
11/9/64	696	433	10,000	2	Water	1.44	0.52	0.98	2.7
12/22/64	739	476	10,000	25	Water	—	—	—	—
1/12/65	760	497	10,000	25	Water	4.58	2.92	1.52	2.9
3/12/65	819	556	10,000	25	Water	11.4	7.87	2.78	4.4
5/26/65	894	631	10,000	25	Water	22.6	14.3	5.03	6.3
8/6/65	966	703	10,000	25	Water	29.2	17.3	6.82	6.9
4/15/66	1218	955	10,000	25	Water	44.8	23.4	10.9	6.5
7/13/66	1307	1044	10,000	25	Water	49.7	24.9	12.4	6.4
10/27/66	1413	1150	10,000	25	Water	54.6	26.4	13.7	6.2

Table 3c
Test Results for Sample 3 of the First Set of Samples

Date	Total Time (days)	Time Under Pressure (days)	Hydrostatic Pressure (psig)	Temp. (°C)	Medium	Density Increase (%)	Volume Decrease (%)	Water Absorption Percentage by Weight	
								Increase (%)	Avg. Increase (%/yr/ 10^4 psig)
12/17/62	0	0	10,000	2	Water	—	—	—	—
2/8/63	53	53	10,000	2	Water	0.56	-0.09	0.68	—
4/16/63	120	120	10,000	2	Water	1.26	-0.31	1.67	—
6/13/63	178	178	10,000	2	Water	1.54	-0.35	1.98	—
8/21/63	247	247	10,000	2	Water	2.66	-0.09	2.78	—
10/15/63	302	302	10,000	2	Water	5.05	+0.18	4.89	—
12/16/63	364	364	10,000	2	Water	6.45	0.57	5.88	5.9
12/17/63	365	364	0	25	Air	—	—	4.95	—
1/21/64	401	364	0	25	Air	—	—	2.72	—
8/14/64	607	364	0	25	Air	0.98	0.00	1.11	—
8/14/64	607	364	10,000	25	Water	—	—	—	—
10/9/64	663	420	10,000	25	Water	18.1	0.13	18.1	111.0
12/4/64	719	476	10,000	25	Water	22.6	0.18	22.4	69.4
2/17/65	794	551	10,000	25	Water	28.2	0.26	27.8	52.1
4/15/65	851	608	10,000	25	Water	32.1	0.09	32.0	46.3
6/11/65	908	665	10,000	25	Water	36.3	0.20	36.1	42.5
2/18/66	1160	917	10,000	25	Water	46.9	0.36	46.4	29.9
2/28/66	1170	927	0	25	Air	—	—	39.6	—

Table 3d
Test Results for Sample 4 of the First Set of Samples

Date	Total Time (days)	Time Under Pressure (days)	Hydrostatic Pressure (psig)	Temp. (°C)	Medium	Density Increase (%)	Volume Decrease (%)	Water Absorption Percentage by Weight	
								Increase (%)	Avg. Increase (%/yr/10^4 psig)
5/23/63	0	0	10,000	2	Water	—	—	—	—
7/29/63	67	67	10,000	2	Water	1.83	0.13	1.68	—
10/10/63	140	140	10,000	2	Water	2.54	0.22	2.43	—
1/13/64	235	235	10,000	2	Water	3.52	0.40	3.11	—
2/27/64	280	280	10,000	2	Water	3.81	0.31	3.54	—
4/16/64	329	329	10,000	2	Water	4.23	0.31	3.98	—
5/26/64	369	369	10,000	2	Water	4.66	0.49	4.17	5.0
5/28/64	371	369	0	25	Air	—	—	3.48	—
8/14/64	449	369	0	25	Air	1.55	0.22	1.37	—
8/14/64	449	369	10,000	25	Water	—	—	—	—
10/9/64	505	425	10,000	25	Water	6.63	0.44	6.16	31.2
12/4/64	561	481	10,000	25	Water	11.7	0.53	11.1	29.6
2/17/65	636	556	10,000	25	Water	15.8	0.49	15.1	26.7
4/15/65	693	613	10,000	25	Water	18.5	0.46	18.0	24.8
6/11/65	750	670	10,000	25	Water	21.3	0.51	20.7	23.3
2/18/66	1002	922	10,000	25	Water	29.5	0.52	28.8	18.1
2/28/66	1012	932	0	25	Air	—	—	22.7	—

Table 3e
Test Results for Sample 5 of the First Set of Samples

Date	Total Time (days)	Time Under Pressure (days)	Hydrostatic Pressure (psig)	Temp. (°C)	Medium	Density Increase (%)	Volume Decrease (%)	Water Absorption Percentage by Weight	
								Increase (%)	Avg. Increase (%/yr/10^4 psig)
5/23/63	0	0	10,000	2	Water	—	—	—	—
7/29/63	67	67	10,000	2	Water	1.69	0.09	1.74	9.5
10/10/63	140	140	10,000	2	Water	2.39	0.13	2.42	6.3
1/13/64	235	235	10,000	2	Water	3.51	0.49	3.16	4.9
2/27/64	280	280	10,000	2	Water	3.37	0.26	3.54	4.6
4/16/64	329	329	10,000	2	Water	4.08	0.26	3.97	4.4
5/26/64	369	369	10,000	2	Water	4.50	0.26	4.22	4.2
5/28/64	371	369	0	25	Air	—	—	3.54	—
9/4/64	470	369	0	25	Air	1.27	0.00	1.30	—
9/4/64	470	369	10,000	2	Water	—	—	—	—
11/9/64	536	435	10,000	2	Water	3.23	0.09	3.23	10.7
12/22/64	579	478	10,000	25	Water	—	—	—	—
1/12/65	600	499	10,000	25	Water	4.50	0.18	4.28	8.4
3/12/65	659	558	10,000	25	Water	6.32	0.13	5.97	9.0
5/26/65	734	633	10,000	25	Water	11.5	0.53	11.0	13.4
8/6/65	806	705	10,000	25	Water	15.9	0.52	15.4	15.3
4/15/66	1058	957	10,000	25	Water	24.9	0.64	24.0	14.1
7/13/66	1147	1046	10,000	25	Water	28.7	0.58	27.9	14.3
10/27/66	1253	1152	10,000	25	Water	32.9	0.52	32.2	14.4

Table 4a
Test Results for Sample 6, the First Sample of the Second Set

Date	Exposure Period (days)	Hydrostatic Pressure (psig)	Temp. (°C)	Medium	Density Increase (%)	Volume Decrease (%)	Water Absorption Percentage by Weight	
							Increase (%)	Avg. Increase (%/yr/ 10^4 psig)
11/29/63	0	10,000	2	Water	—	—	—	—
2/11/64	74	10,000	2	Water	0.55	-0.09	0.85	4.2
4/10/64	133	10,000	2	Water	1.65	0.00	1.70	4.7
6/9/64	193	10,000	2	Water	2.34	0.00	2.50	4.7
7/27/64	241	10,000	2	Water	2.89	0.00	3.04	4.6
9/24/64	300	10,000	2	Water	3.44	-0.04	3.59	4.4
12/4/64	371	10,000	2	Water	4.13	-0.09	4.38	4.3
12/22/64	389	10,000	25	Water	—	—	—	—
2/9/65	438	10,000	25	Water	5.79	-0.04	6.03	5.0
2/24/65	453	10,000	25	Water	6.48	-0.09	6.44	5.2
4/12/65	500	10,000	25	Water	7.16	-0.26	7.40	5.4
6/22/65	571	10,000	25	Water	8.54	-0.01	8.72	5.6
3/25/66	847	10,000	25	Water	12.0	-0.01	12.1	5.2
7/7/66	951	10,000	25	Water	14.0	+0.13	14.1	5.4
10/20/66	1056	10,000	25	Water	15.7	-0.10	15.9	5.5
3/2/67	1189	10,000	25	Water	17.9	-0.12	18.2	5.9

Table 4b
Test Results for Sample 7, the Second Sample of the Second Set

Date	Exposure Period (days)	Hydrostatic Pressure (psig)	Temp. (°C)	Medium	Density Increase (%)	Volume Decrease (%)	Water Absorption Percentage by Weight	
							Increase (%)	Avg. Increase (%/yr/ 10^4 psig)
11/29/63	0	10,000	25	Water	—	—	—	—
2/4/64	67	10,000	25	Water	1.67	+0.04	1.54	8.4
3/19/64	111	10,000	25	Water	2.51	-0.09	2.52	8.3
6/1/64	185	10,000	25	Water	5.02	+0.04	4.87	9.6
7/17/64	231	10,000	25	Water	5.57	-0.09	5.61	8.9
9/17/64	293	10,000	25	Water	7.24	-0.09	7.33	9.1
11/27/64	364	10,000	25	Water	8.91	-0.09	9.00	9.0
1/14/65	412	10,000	25	Water	10.3	0.00	10.4	9.2
3/22/65	479	10,000	25	Water	11.7	-0.18	11.8	9.0
5/18/65	536	10,000	25	Water	13.2	-0.06	13.3	9.0
3/16/66	838	10,000	25	Water	18.4	-0.08	18.4	8.0
6/29/66	943	10,000	25	Water	20.9	+0.04	21.0	8.1
10/5/66	1041	10,000	25	Water	22.6	-0.45	23.0	8.1
2/27/67	1186	10,000	25	Water	25.5	-0.23	25.7	8.3

Table 4c
Test Results for Sample 8, the Third Sample of the Second Set

Date	Exposure Period (days)	Hydrostatic Pressure (psig)	Temp. (°C)	Medium	Density Increase (%)	Volume Decrease (%)	Water Absorption Percentage by Weight	
							Increase (%)	Avg. Increase (%/yr/ 10^4 psig)
11/29/63	0	10,000	2	Water	—	—	—	—
2/11/64	74	10,000	2	Water	0.41	-0.04	0.55	2.7
4/10/64	133	10,000	2	Water	0.96	0.00	1.04	2.9
6/9/64	193	10,000	2	Water	1.52	+0.09	1.53	2.9
7/27/64	241	10,000	2	Water	1.65	-0.09	1.90	2.9
9/24/64	300	10,000	2	Water	2.20	+0.04	2.33	2.8
12/4/64	371	10,000	2	Water	2.48	0.00	2.58	2.5
12/22/64	389	10,000	25	Water	—	—	—	—
2/9/65	438	10,000	25	Water	3.31	-0.13	3.49	2.9
2/24/65	453	10,000	25	Water	3.72	+0.09	3.63	2.9
4/12/65	500	10,000	25	Water	3.99	-0.27	4.09	3.0
6/22/65	571	10,000	25	Water	5.38	+0.16	5.27	3.4
3/25/66	847	10,000	25	Water	7.71	+0.12	7.64	3.3
7/7/66	951	10,000	25	Water	8.95	0.25	8.70	3.3
10/20/66	1056	10,000	25	Water	9.78	0.17	9.62	3.3
3/2/67	1189	10,000	25	Water	10.9	0.16	10.7	3.5

Table 4d
Test Results for Sample 9, the Fourth Sample of the Second Set

Date	Exposure Period (days)	Hydrostatic Pressure (psig)	Temp. (°C)	Medium	Density Increase (%)	Volume Decrease (%)	Water Absorption Percentage by Weight	
							Increase (%)	Avg. Increase (%/yr/ 10^4 psig)
11/29/63	0	10,000	25	Water	—	—	—	—
2/4/64	67	10,000	25	Water	0.97	0.00	1.48	8.1
3/19/64	111	10,000	25	Water	1.67	0.04	2.10	6.9
6/1/64	185	10,000	25	Water	2.50	0.22	2.84	5.6
7/17/64	231	10,000	25	Water	3.20	0.04	3.52	5.6
9/17/64	293	10,000	25	Water	4.17	0.22	4.38	5.5
11/27/64	364	10,000	25	Water	5.00	0.22	5.24	5.3
1/14/65	412	10,000	25	Water	5.56	0.22	5.86	5.2
3/22/65	479	10,000	25	Water	6.39	0.18	6.42	4.9
5/18/65	536	10,000	25	Water	7.22	0.28	7.31	4.9
3/16/66	838	10,000	25	Water	9.72	0.32	9.86	4.3
6/29/66	943	10,000	25	Water	11.1	0.40	11.1	4.3
10/5/66	1041	10,000	25	Water	12.2	-0.05	12.8	4.5
2/27/67	1186	10,000	25	Water	13.8	+0.27	13.9	4.5

Table 5 presents data on these last four samples. All test samples will be kept pressurized at room temperature for an indefinite period of time. Figures 3 through 25 are graphs of the data contained in Tables 3 through 5.

Table 5

Test Results Obtained for the Final Four Samples

Date	Total Time (days)	Time Under Pressure (days)	Hydrostatic Pressure (psig)	Temp. (°C)	Medium	Density Increase (%)	Volume Decrease (%)	Water Absorption Percentage by Weight	
								Increase (%)	Avg. Increase (%/yr/10^4psig)
3/7/66*	0	0	5,000	25	Water	—	—	—	—
6/6/66	91	91	5,000	25	Water	1.64	-0.01	1.68	13.5
7/19/66	134	134	5,000	25	Water	2.13	0.00	2.11	11.5
12/28/66	296	296	5,000	25	Water	3.11	+0.07	3.04	7.5
3/21/67	379	379	5,000	25	Water	3.44	-0.08	3.39	6.5
3/7/66†	0	0	5,000	25	Water	—	—	—	—
6/6/66	91	91	5,000	25	Water	1.83	0.08	1.80	14.4
7/19/66	134	134	5,000	25	Water	2.33	0.12	2.17	11.8
12/28/66	296	296	5,000	25	Water	3.49	0.19	3.16	7.8
3/21/67	379	379	5,000	25	Water	3.99	0.03	3.55	6.8
3/11/66‡	0	0	10,000	25	Water	—	—	—	—
6/16/66	97	97	10,000	25	Water	1.60	0.35	1.18	4.4
8/17/66	159	159	10,000	25	Water	2.18	0.17	2.02	4.6
1/4/67	299	299	10,000	25	Water	3.35	-0.04	3.39	4.1
3/11/66§	0	0	10,000	25	Water	—	—	—	—
3/16/66	97	97	10,000	25	Water	1.46	0.33	1.21	4.6
8/17/66	159	159	10,000	25	Water	2.19	0.15	2.18	5.0
1/4/67	299	299	10,000	25	Water	3.36	0.02	3.46	4.2

*Sample 10.
†Sample 11.
‡Sample 12.
§Sample 13.

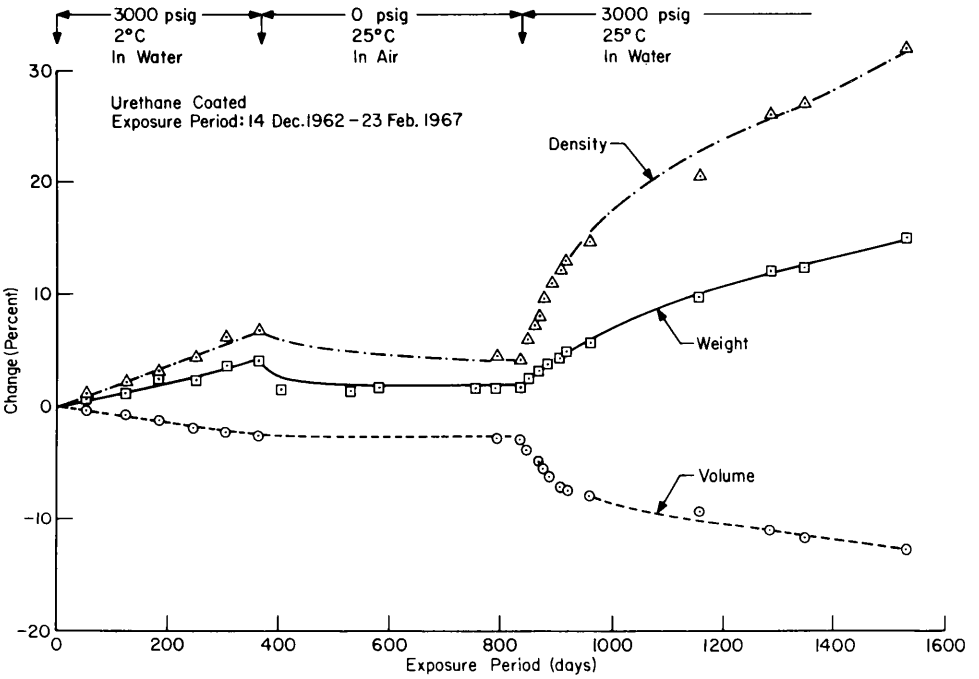

Fig. 3 - Effect of air drying and temperature increase on urethane-coated sample 1

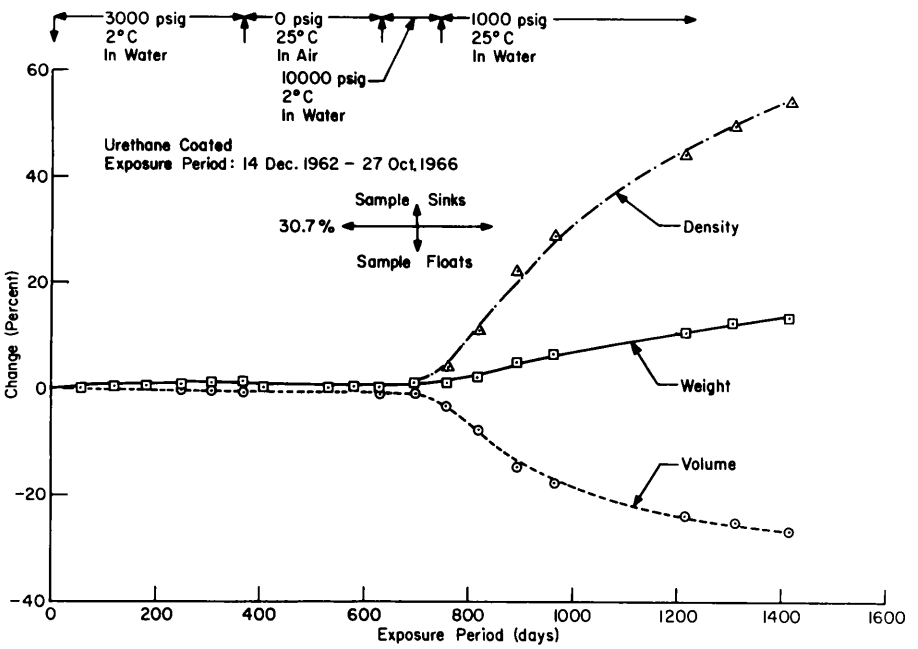

Fig. 4 - Effect of air drying and pressure and temperature increase on urethane-coated sample 2

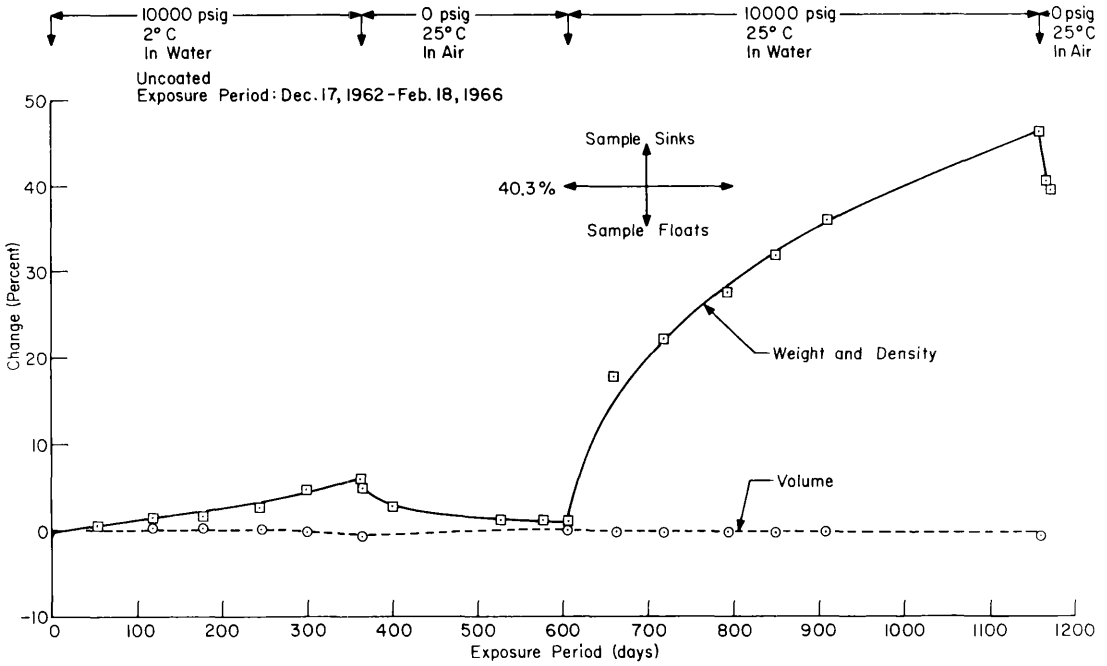

Fig. 5 - Effect of air drying and temperature increase on uncoated sample 3

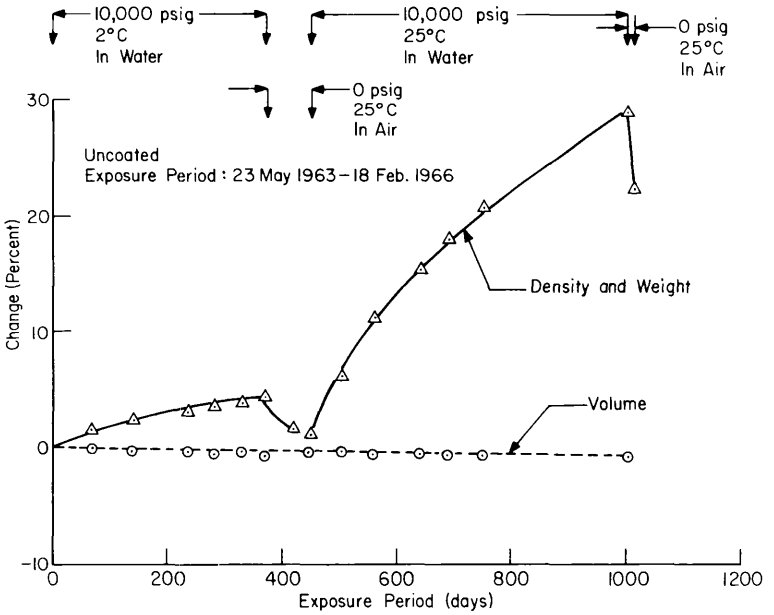

Fig. 6 - Effect of air drying and temperature increase on uncoated sample 4

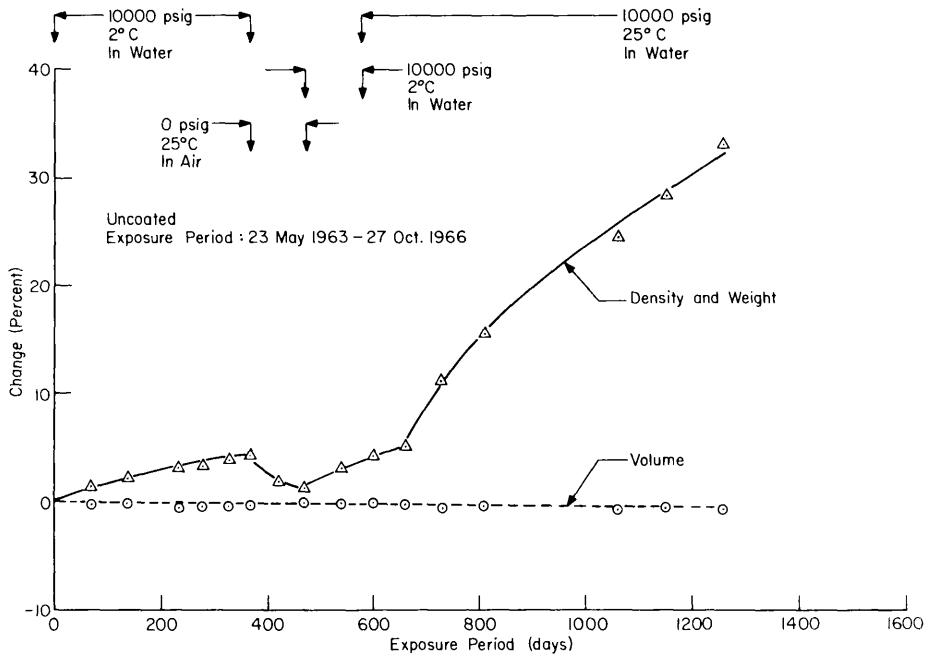

Fig. 7 - Effect of air drying and temperature increase on uncoated sample 5

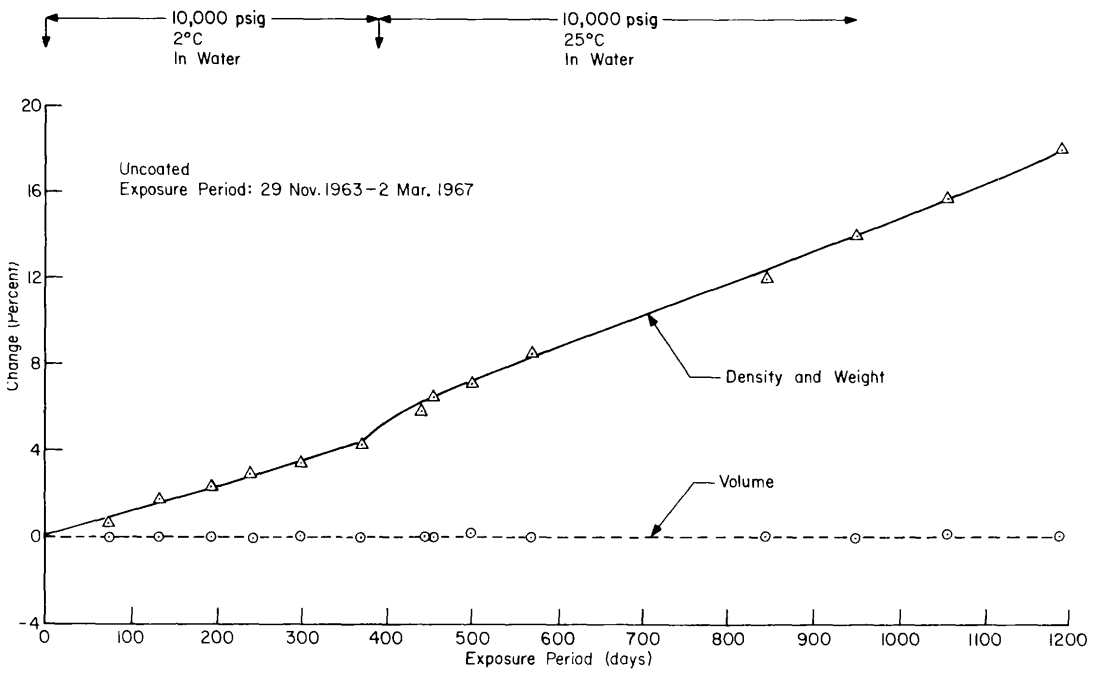

Fig. 8 - Effect of temperature increase on uncoated sample 6

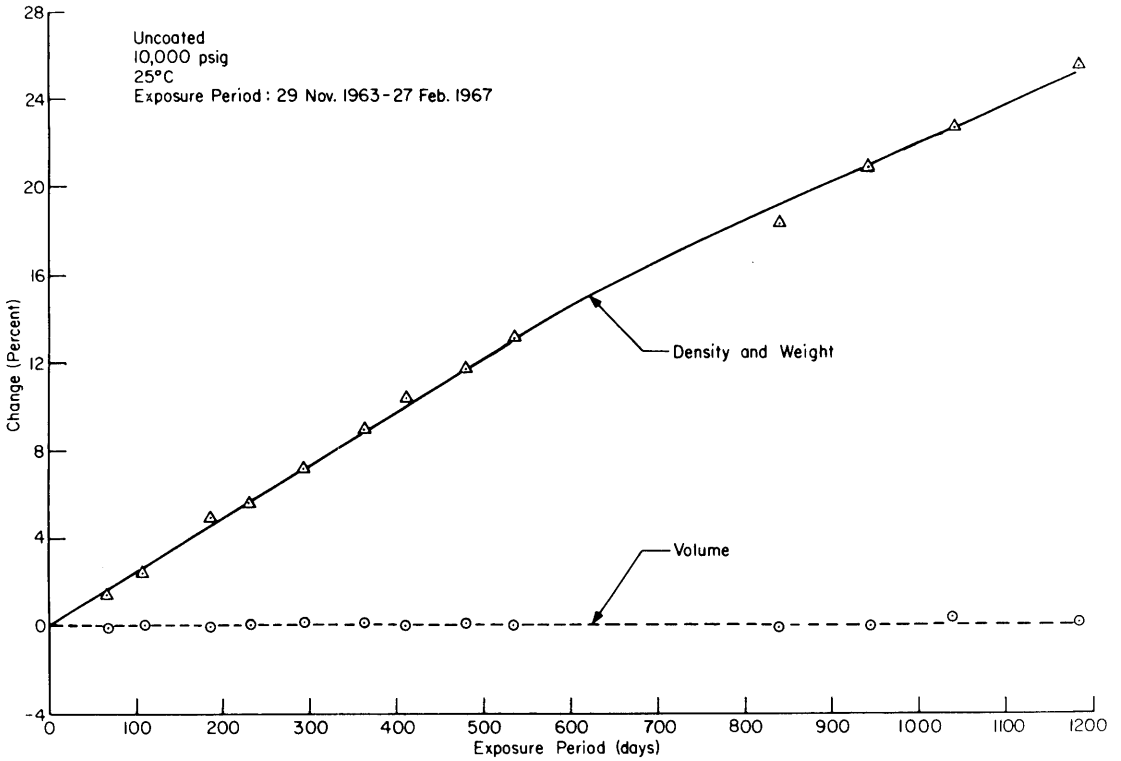

Fig. 9 - Hydrostatic pressure of 10,000 psig on uncoated sample 7

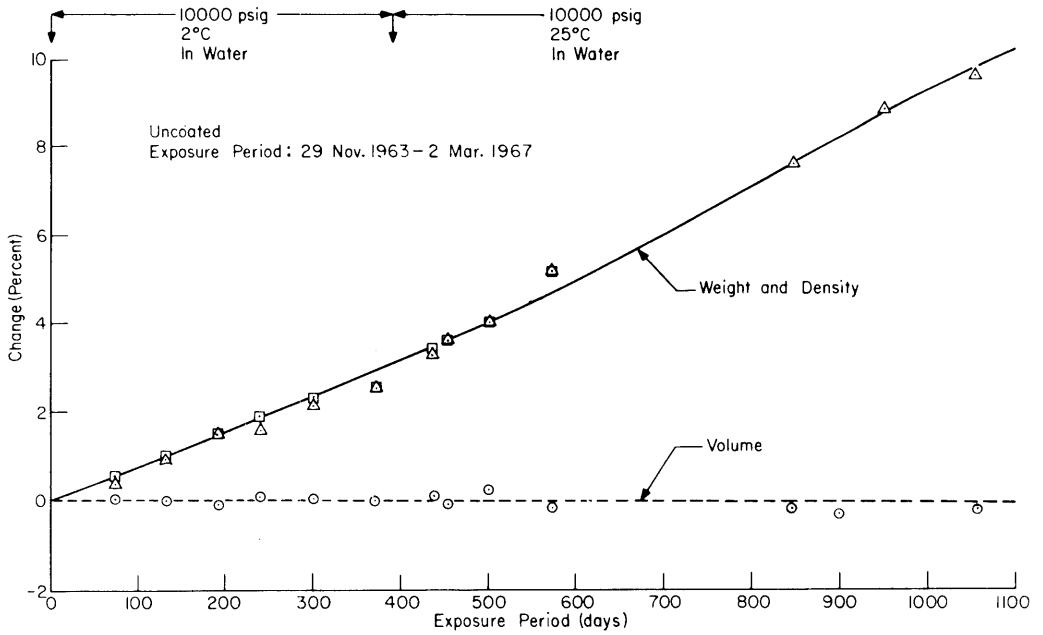

Fig. 10 - Effect of temperature increase on uncoated sample 8

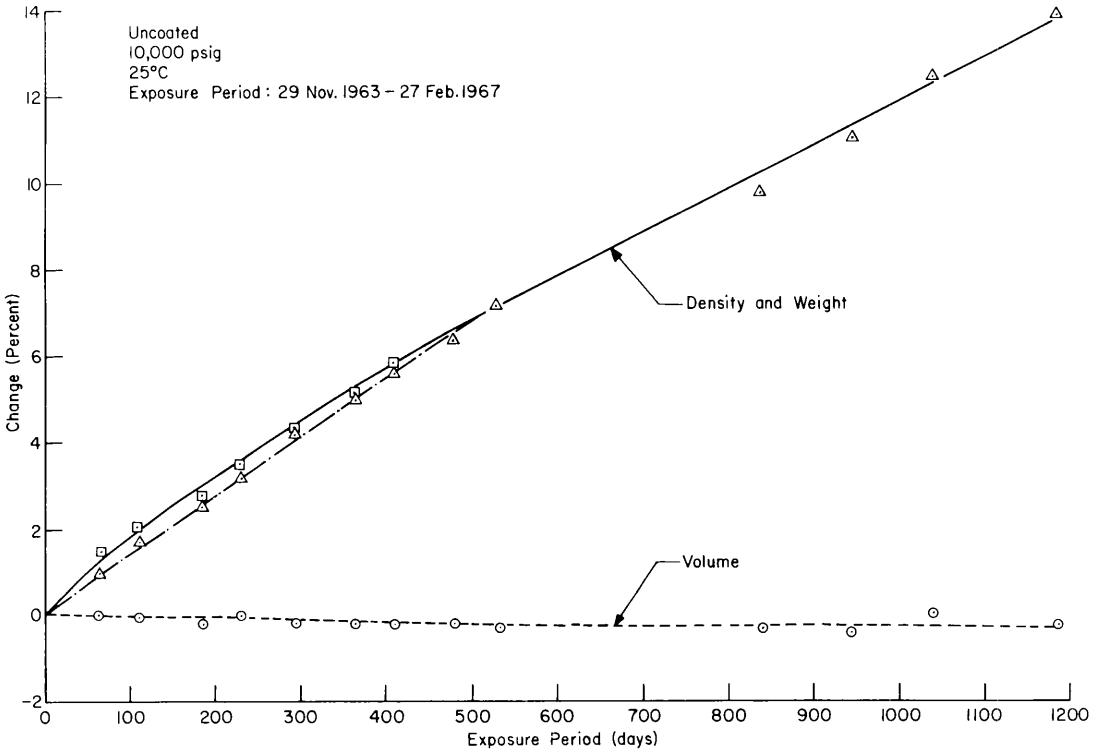

Fig. 11 - Hydrostatic pressure of 10,000 psig on uncoated sample 9

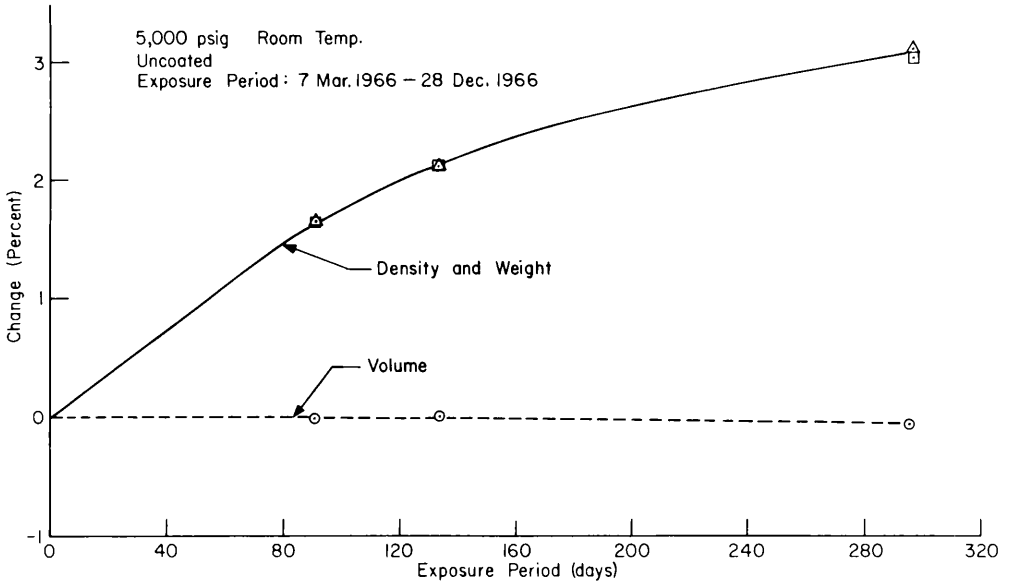

Fig. 12 - Hydrostatic pressure of 5000 psig on uncoated sample 10

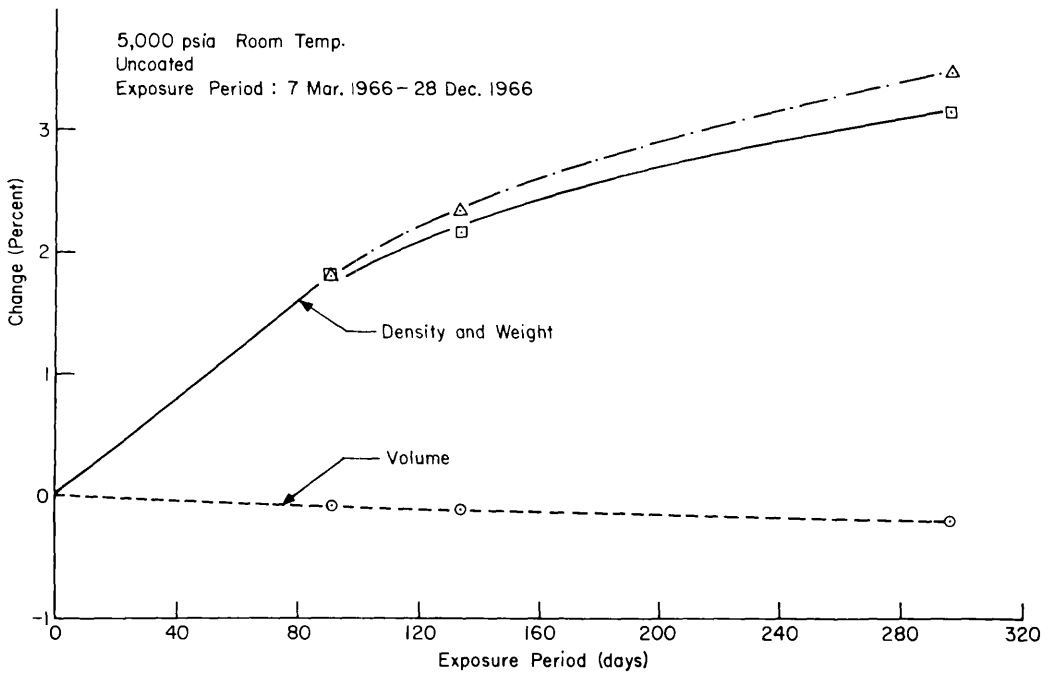

Fig. 13 - Hydrostatic pressure of 5000 psig on uncoated sample 11

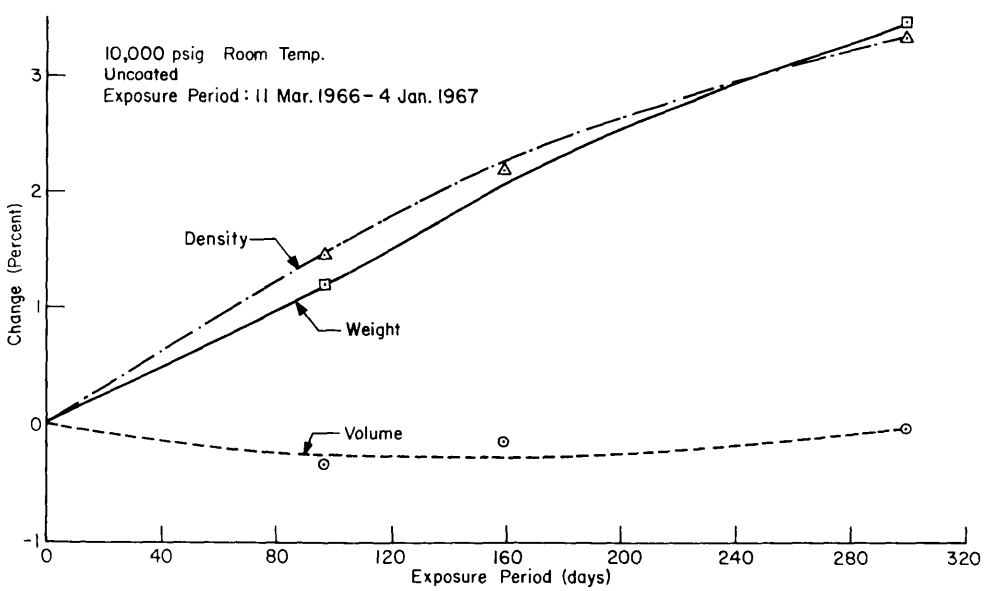

Fig. 14 - Hydrostatic pressure of 10,000 psig on uncoated sample 12

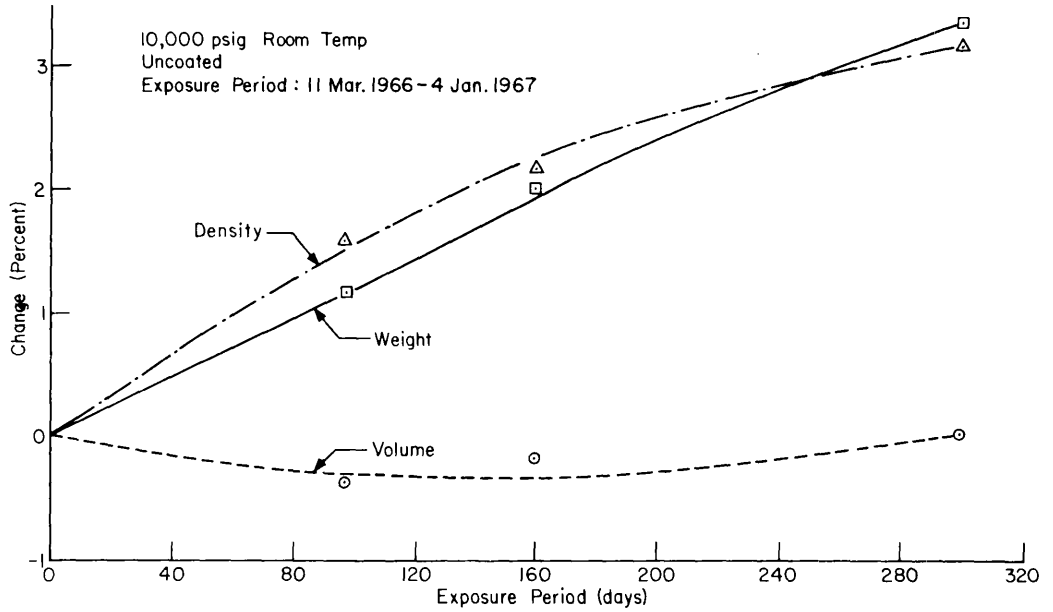

Fig. 15 - Hydrostatic pressure of 10,000 psig on uncoated sample 13

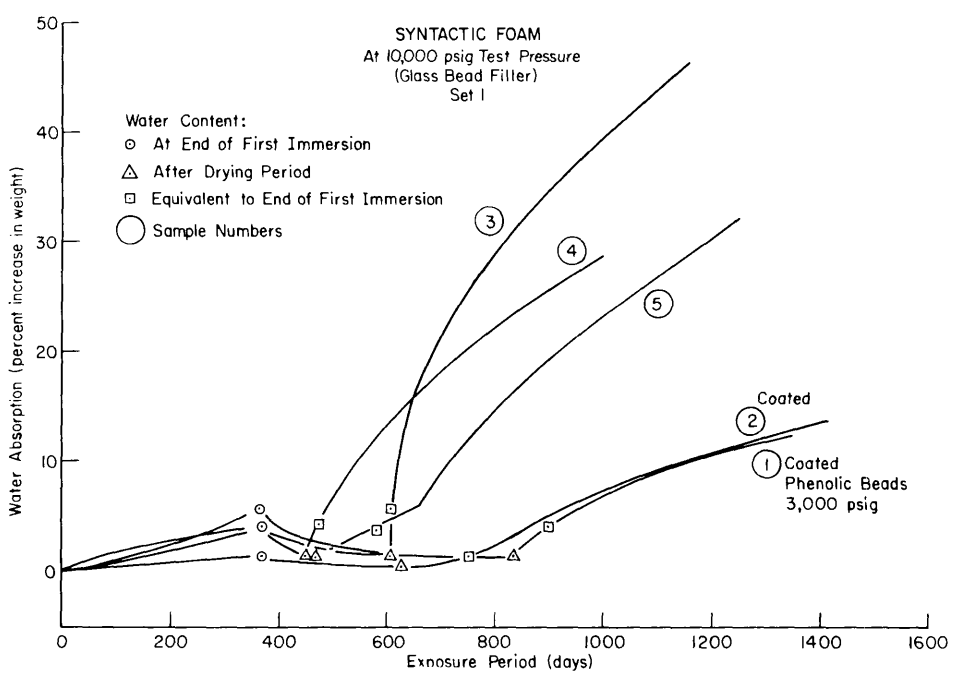

Fig. 16 - Effect of air drying on water content of Set I samples

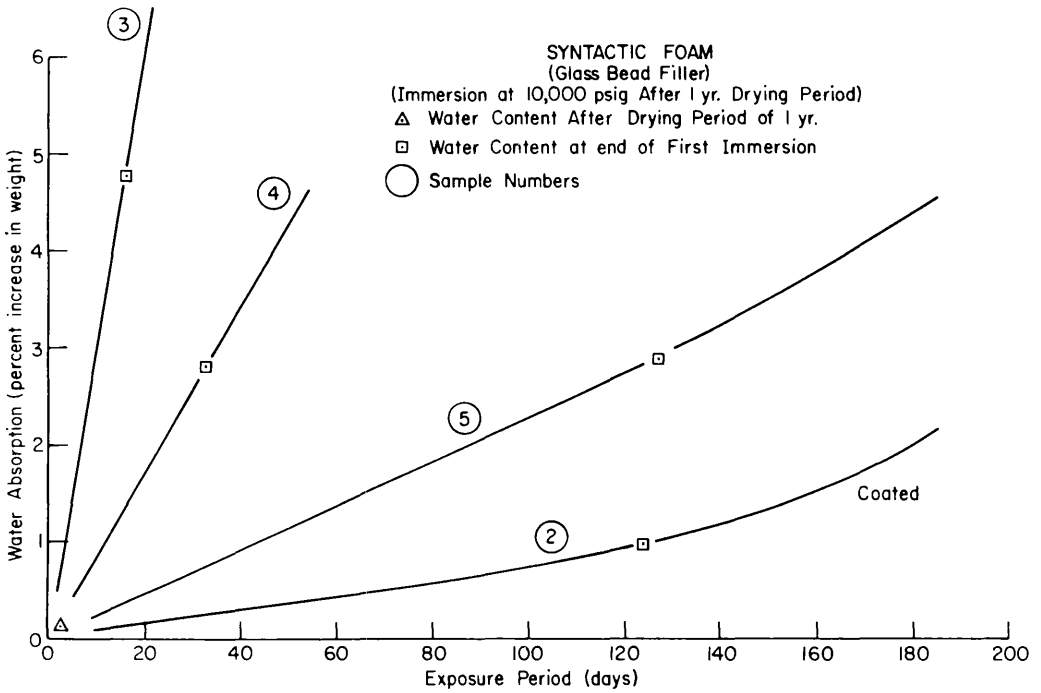

Fig. 17 - Water absorption after air drying of Set 1 samples

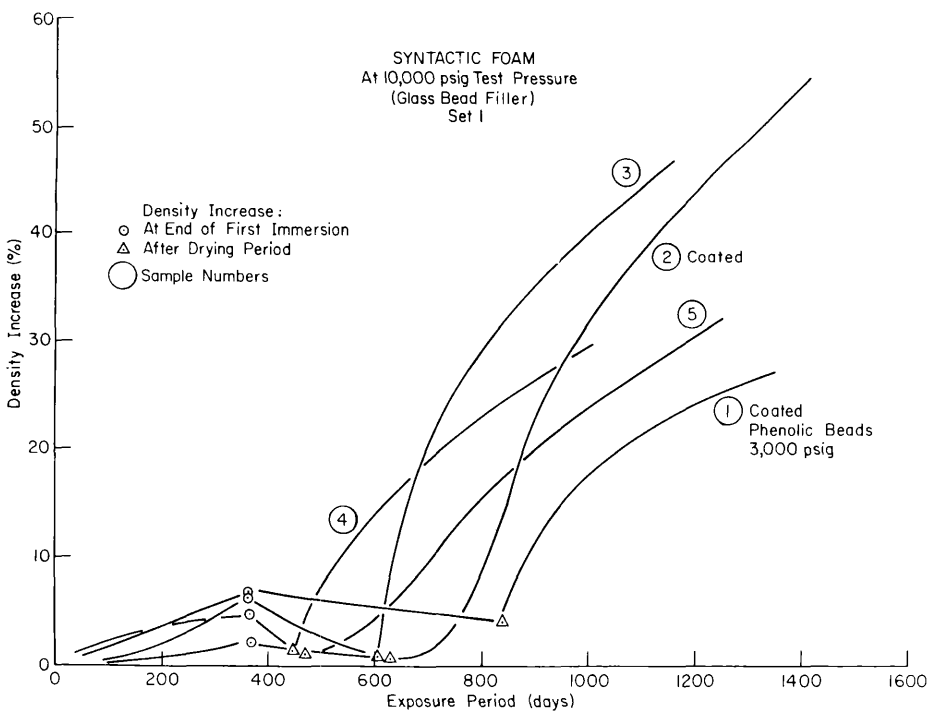

Fig. 18 - Effect of air drying on density increase of Set 1 samples

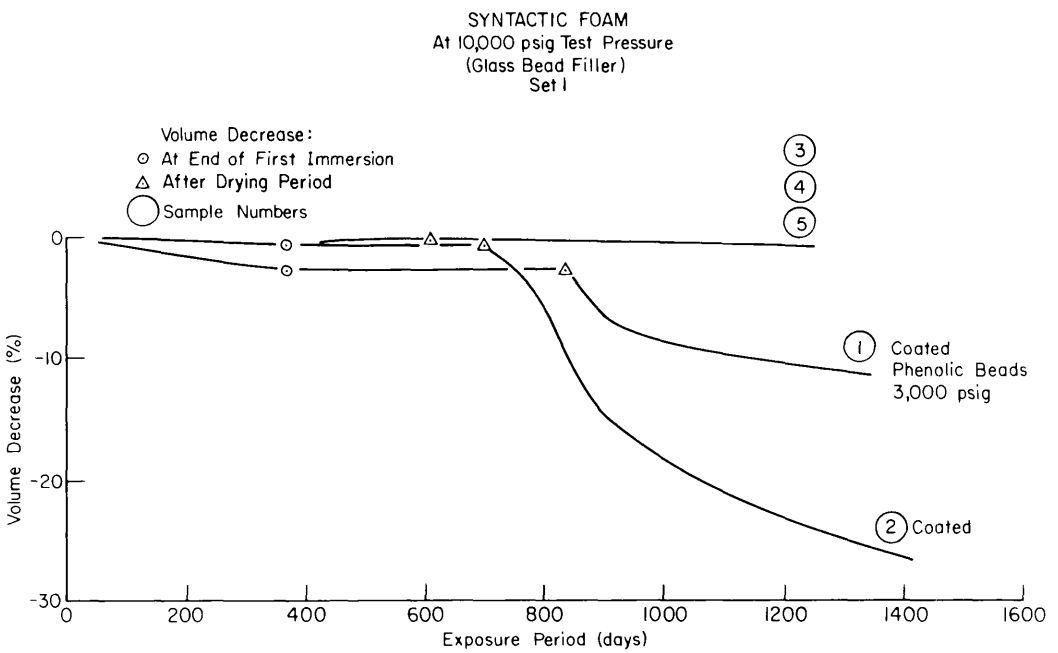

Fig. 19 - Effect of air drying on volume decrease of Set 1 samples

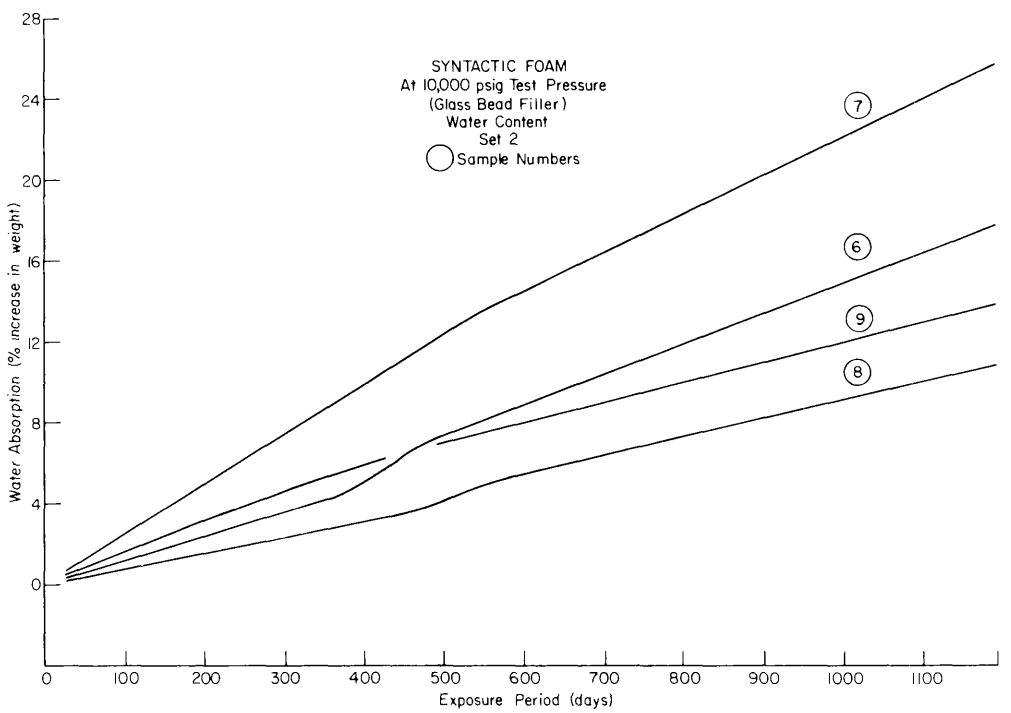

Fig. 20 - Water content increase of Set 2 samples

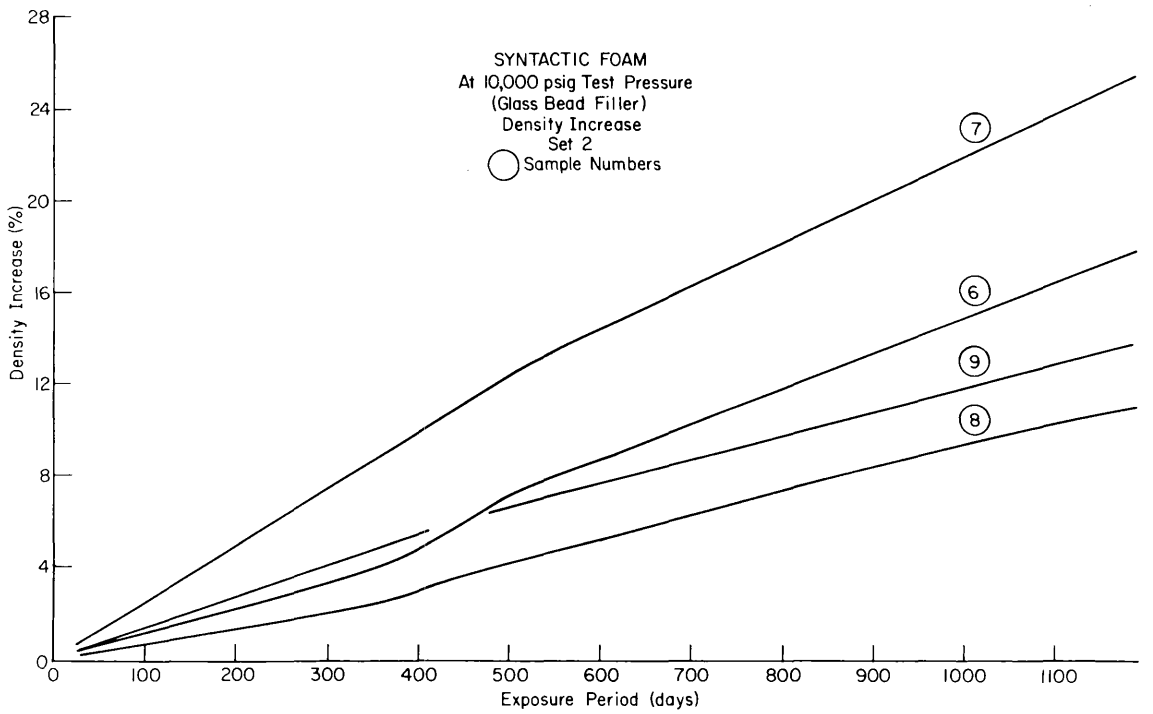

Fig. 21 - Density increase of Set 2 samples

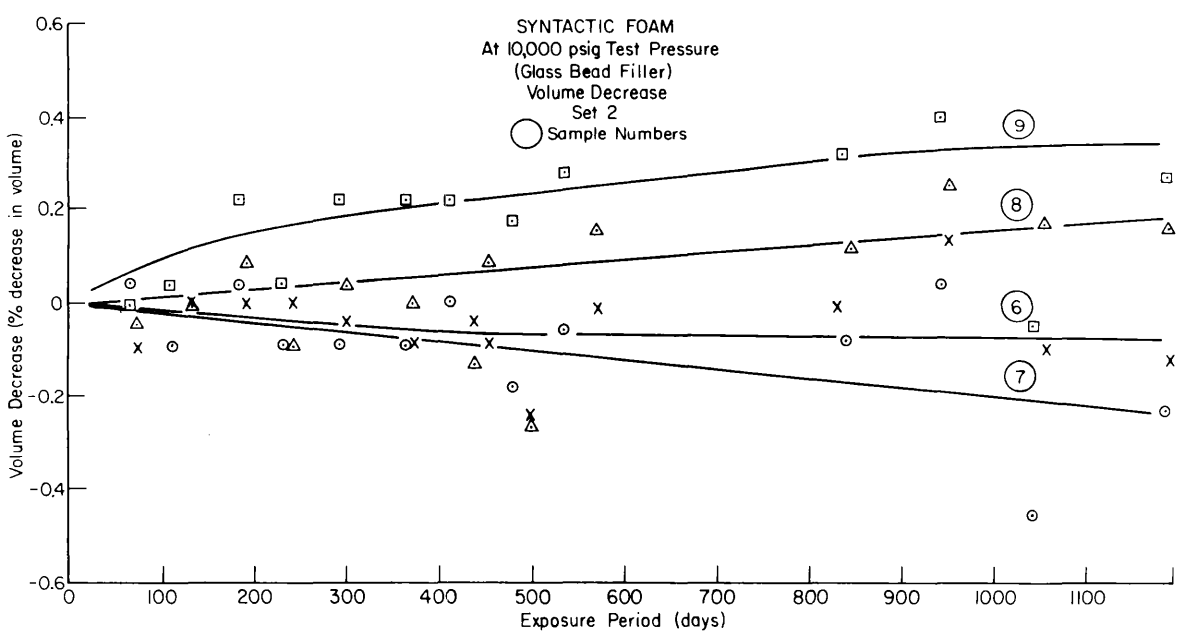

Fig. 22 - Volume change of Set 2 samples

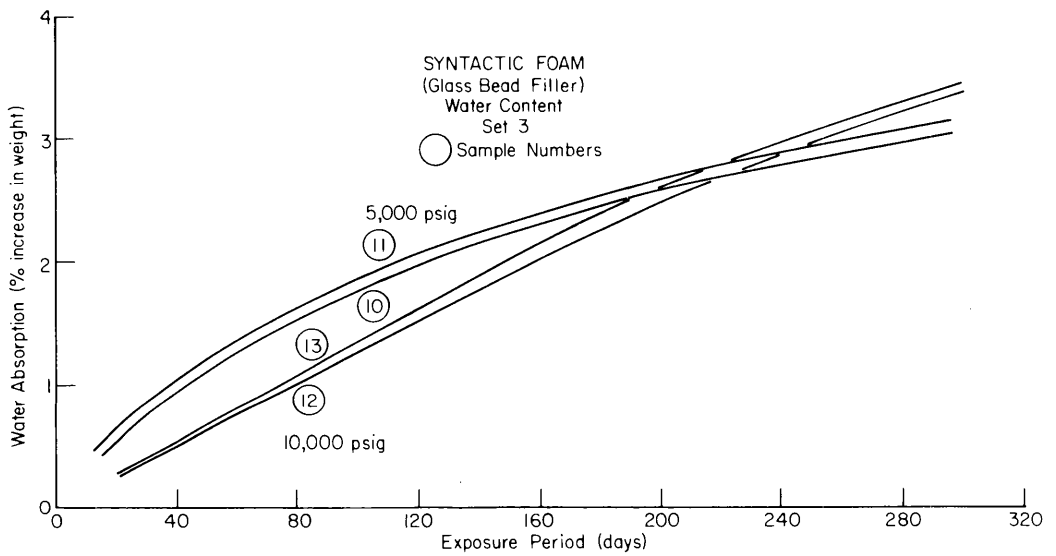

Fig. 23 - Water content increase of Set 3 samples

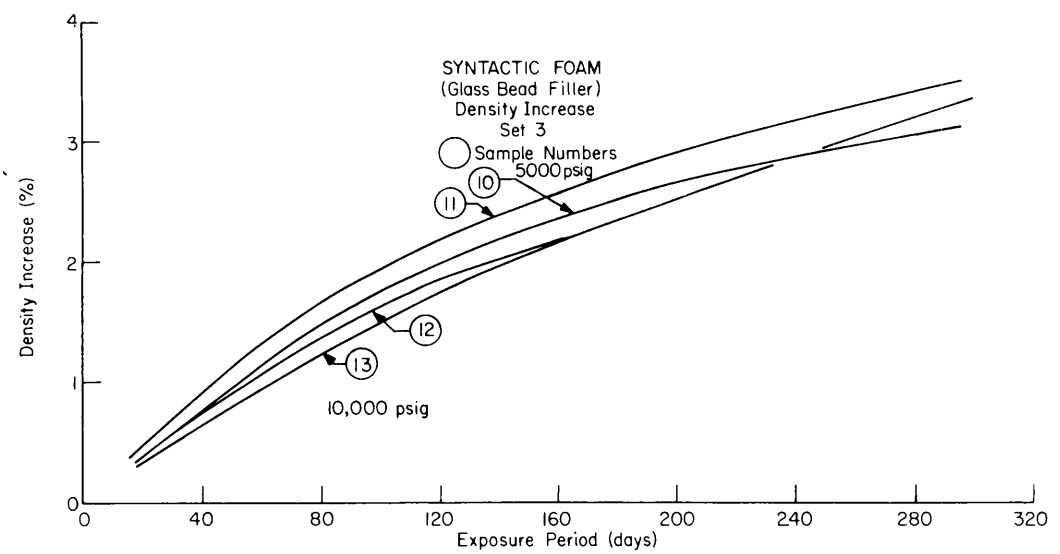

Fig. 24 - Density increase of Set 3 samples

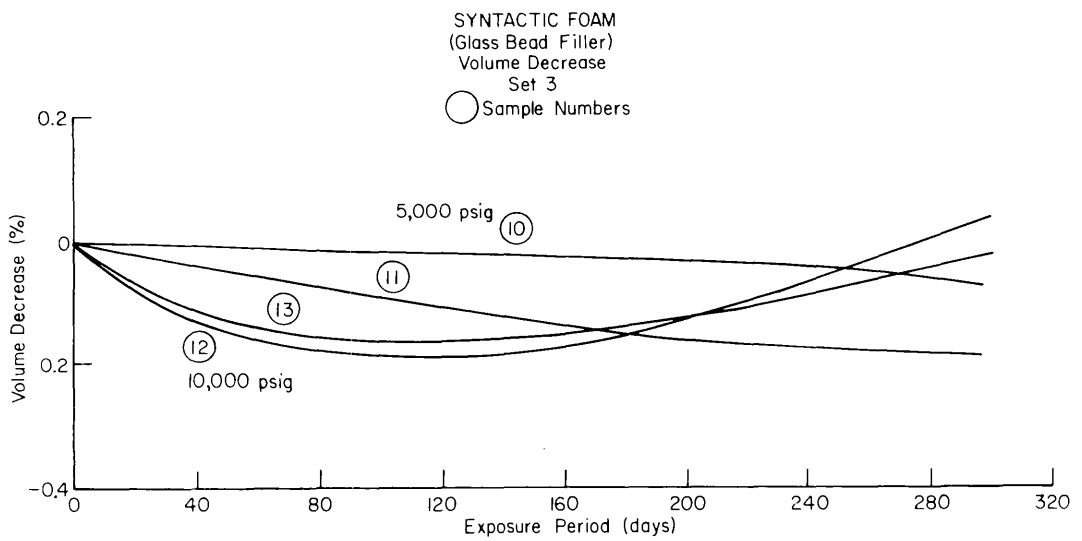

Fig. 25 - Volume change of Set 3 samples

DISCUSSION

It is interesting to note the behavior of the samples in set 1 (Fig. 16) which had been air dried for various periods after having been pressurized for one year and, again, subjected to the same hydrostatic test pressure of 10,000 psig. These samples absorbed water at a more rapid rate after drying than when originally pressurized. In approximately two weeks sample 3 (Fig. 17), and in one month sample 4, had absorbed the same amount of water at 2°C which these uncoated samples had originally absorbed in the previous year of hydrostatic immersion at 2°C. It required four months for samples 2 and 5 to reach this same level of water content as compared to their first year of exposure. This longer period may be due partly to their hydrostatic reexposure at 2°C for the period during which they reached a water content equivalent to their original level. When testing was changed from 2°C to room temperature, their rate of absorption increased (Fig. 16). The water absorption rate for this sample-group, after three years of testing, shows an increase with time.

The second set of samples (6, 7, 8, and 9) were not air dried. These samples are of a better grade of syntactic foam. The water absorption rate is practically linear with time. Sample 8 shows a water pickup rate of 4% per year, and sample 7 shows an 8% rate. These samples have approximately the same density.

Of the third set, samples 10 and 11 have a water pickup rate of 4% per year. Samples 10 and 11 were at 5000 psi and have a density 37.7 lb/ft^3. Samples 12 and 13, exposed to 10,000 psig, have a water pickup rate of 4.5% per year and a density of 42.4 lb/ft^3.

An understanding of the water pickup method of syntactic foam is needed. There are several hypotheses with some supporting evidence which lend partial explanations. There is evidence from other experimenters that water diffuses into the foam as a result of decompression from the test pressure to atmospheric conditions — similar to the action of a sponge. In this event, the water pickup would be of no detrimental significance, since it would not alter the buoyancy of the foam when under pressure.

The data presented, however, presses for a different conclusion. Why does the water pickup amount increase with time? If it were purely sponge-action, the amount of water absorbed from the same pressurized condition to atmospheric should remain fairly constant with time. It is the authors' opinion that there is progressive rupture of the glass beads from the surface inward which is directly related to time. Given sufficient time, enough glass beads will rupture, and the float will sink. The tests conducted indicate this premise to be true. Samples 2 and 3, after having picked up sufficient water, did in fact sink (when checked at atmospheric conditions). These samples were sliced in half and etched (Figs. 26 and 27). The red dye penetrant is believed to depict the areas of broken glass beads resulting from hydrostatic pressure. By reflected light it was not optically possible to distinguish broken beads in this area as being different in appearance from the broken beads in other areas caused by slicing the sample in half. Figure 28 is a magnified view of small areas of samples 2 and 3. However, by transmitting light through the sections, photomicrographs were obtained (Figs. 29 through 32). The four (75 magnification) photomicrographs show areas in sequence from the outer edge to the inner portion of the sample, the dark disk in Figs. 30 and 31 in the same area as was outlined by the dye penetrant (Figs. 26 and 27). No analysis is offered for the darkening phenomenon at this time.

To determine whether the glass beads in the outer sections of the sample had indeed been broken, one of the samples was sliced into eight sections and each section was floated in water. If the outermost pieces should sink while the pieces taken from the central part of the sample did not sink – a conclusion may be drawn. To test this hypothesis, the sample was divided into sections representing various conditions in the material. Figure 33 shows the precise manner in which the sample was sliced.

The slices were subjected to a hydrostatic pressure of 3000 psi for one hour and were then placed in the water-filled graduated glass tube shown in Fig. 34. Table 6 lists pertinent data concerning the segments of the sample. As tabulated and as shown in the photograph, the outer slices sank while the centrally selected slices floated. Based on this evidence, it is concluded that the glass beads depicted by the dye penetrant are ruptured.

CONCLUSIONS

Based on the data presented, the following observations are made:

1. Water absorption is a function of pressure, density, and temperature -- in this order of importance.

2. Water absorption of a pressurized sample increases almost linearly with time of exposure.

3. Progressive water pickup by hydraulically pressurized syntactic foam is caused by water entering the cavities of ruptured glass beads. As the more inboard glass beads continue to rupture and fill with water, the float will eventually sink.

4. Drying the samples does not restore the material to its original buoyancy capability.

5. At the present time the life expectancy (no longer buoyant) of a good grade of 42-lb/ft^3 syntactic foam at 10,000 psig is estimated to be about 10 years. Table 7 shows the calculated life expectancy of the syntactic foams under test. As is indicated, the life expectancy of the foam is extended as the technology of manufacture continues to improve. The samples represent the technology of the past four years.

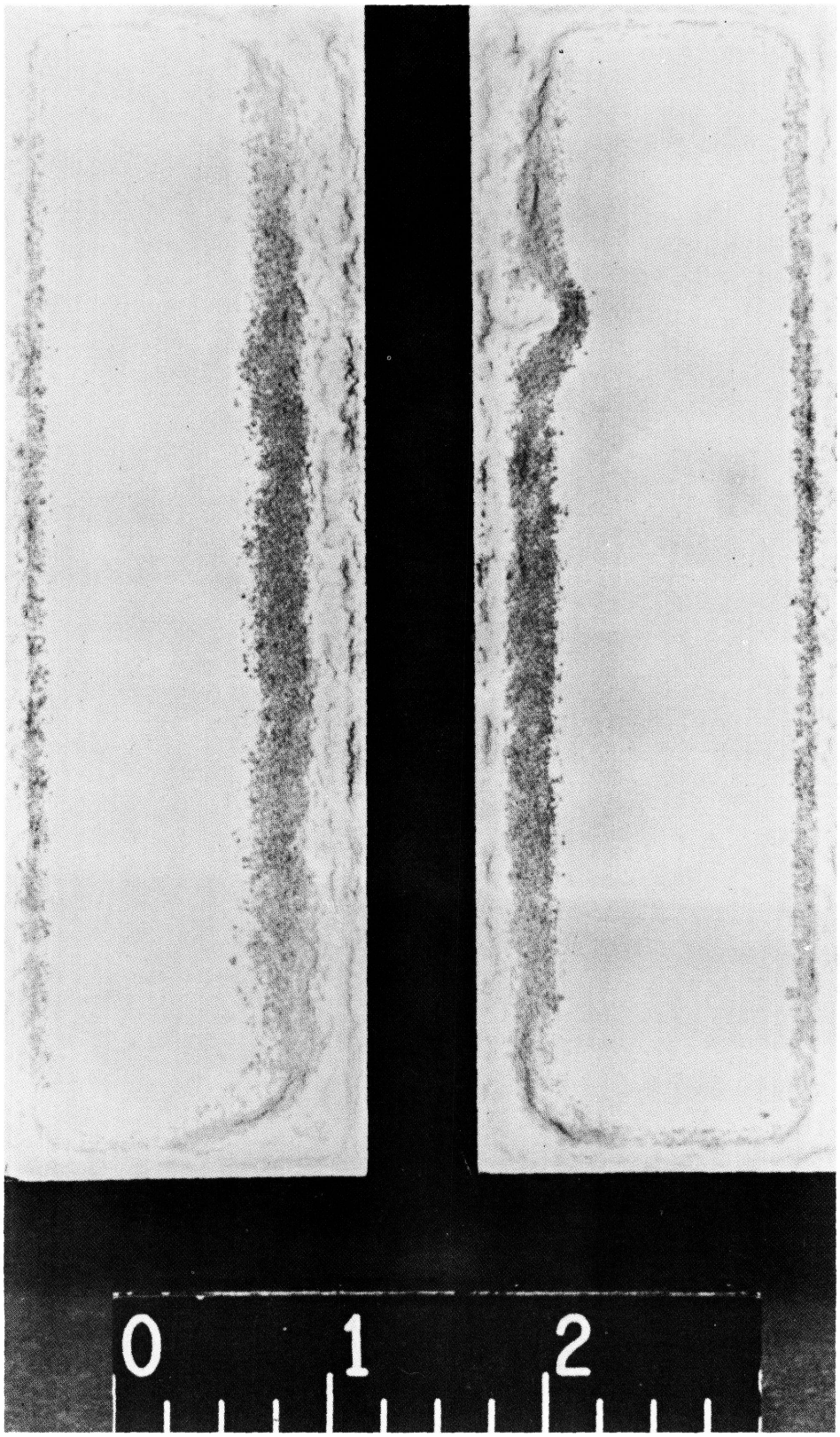

Fig. 26 - Extent of water penetration of sample 3

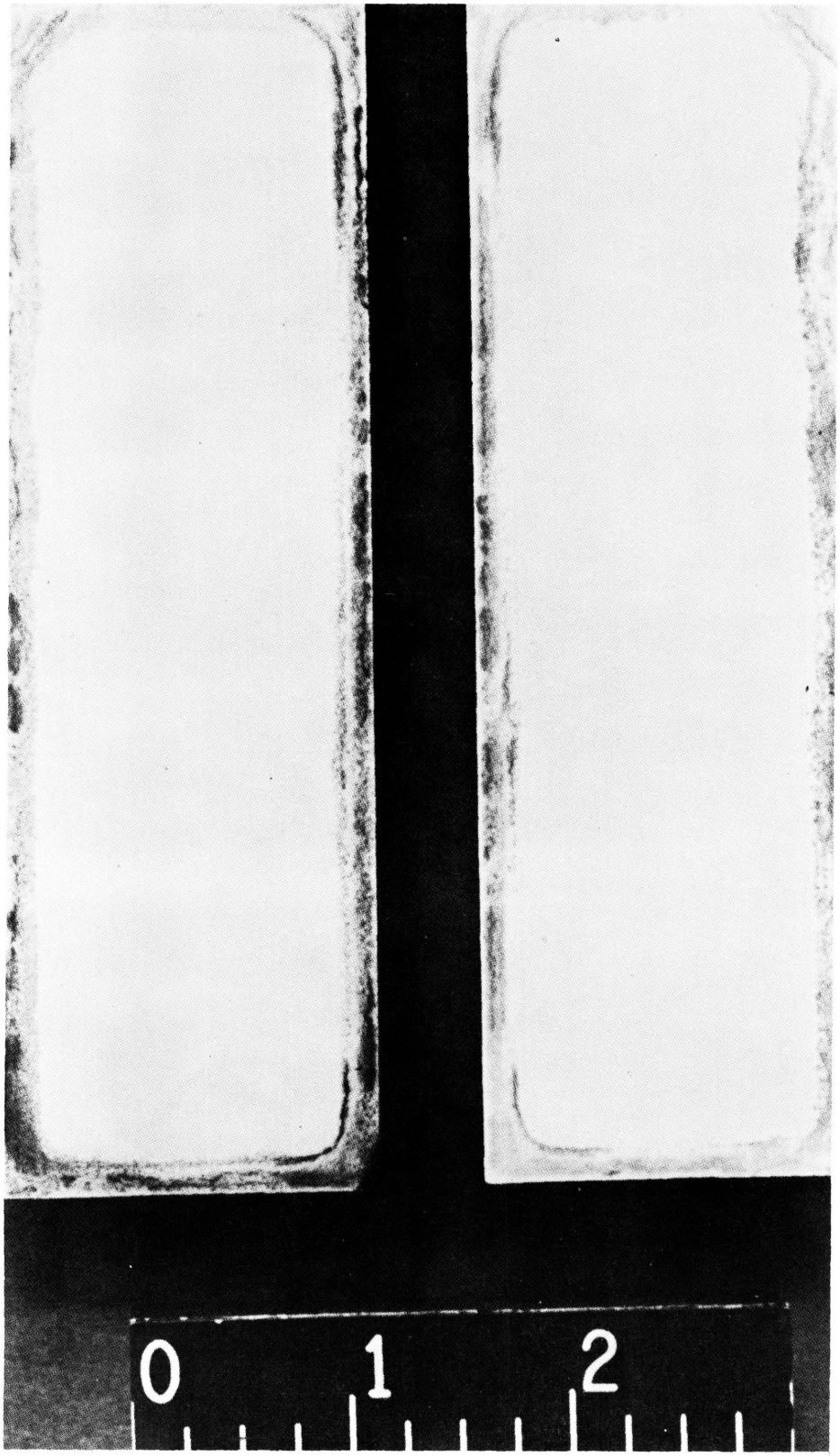

Fig. 27 - Extent of water penetration of sample 4

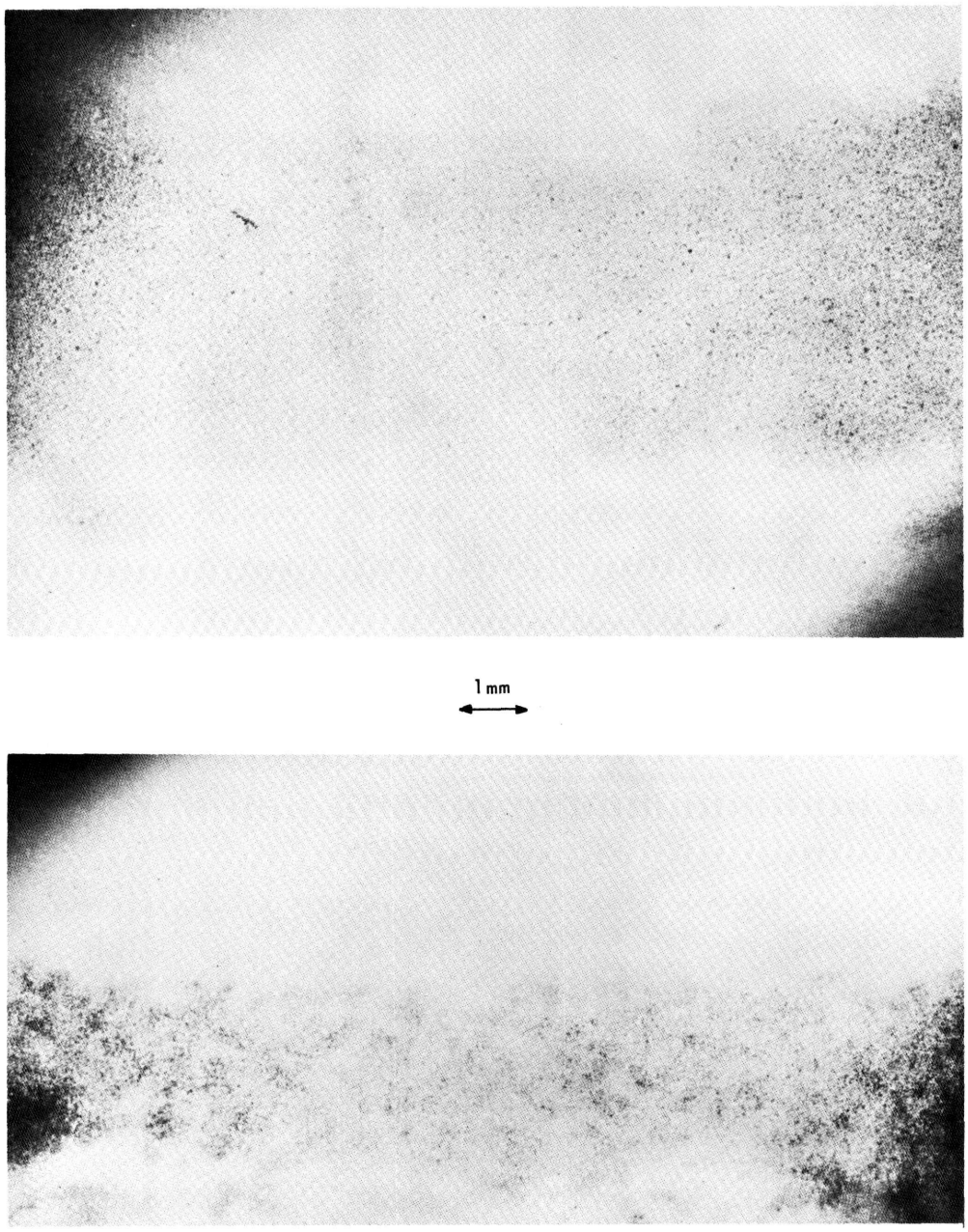

Fig. 28 - Broken beads area of samples 3 and 4 (10X)

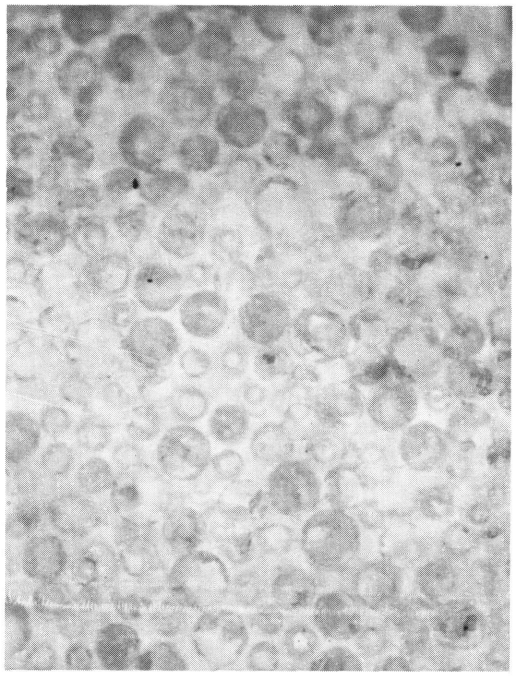

Fig. 29 - Extreme edge of the sample under 4-min exposure with green filter

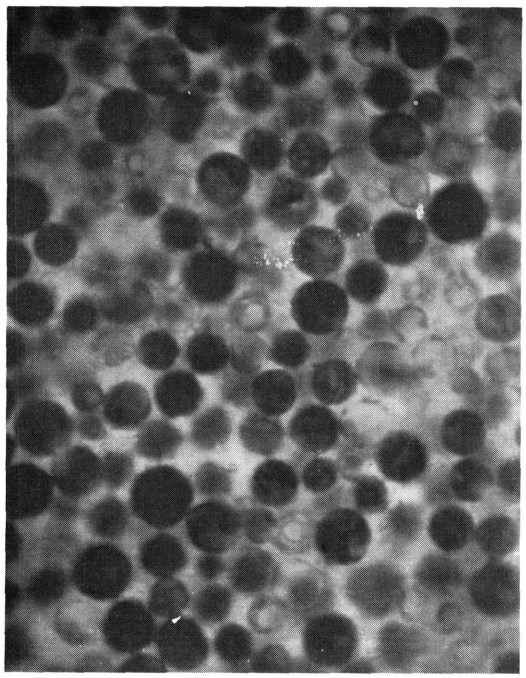

Fig. 30 - White or broken area under 3-min exposure with green filter

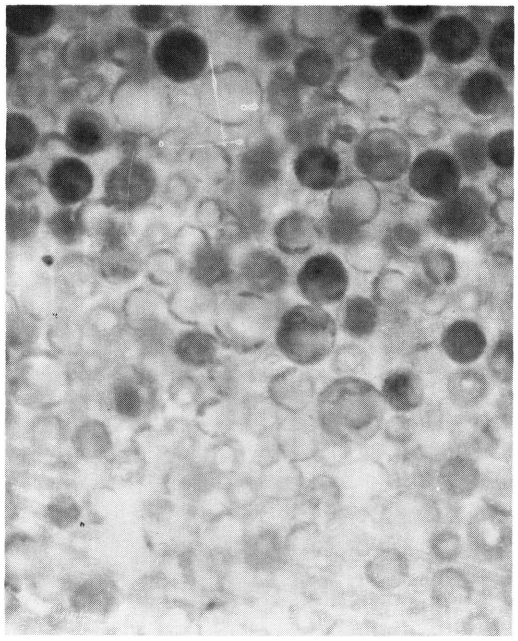

Fig. 31 - Boundary between white or broken area and center section; 2 min, green filter

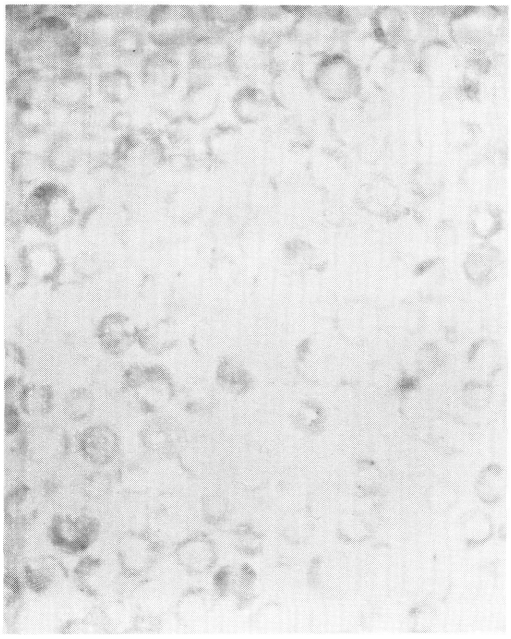

Fig. 32 - Center section; 2-min exposure, green filter

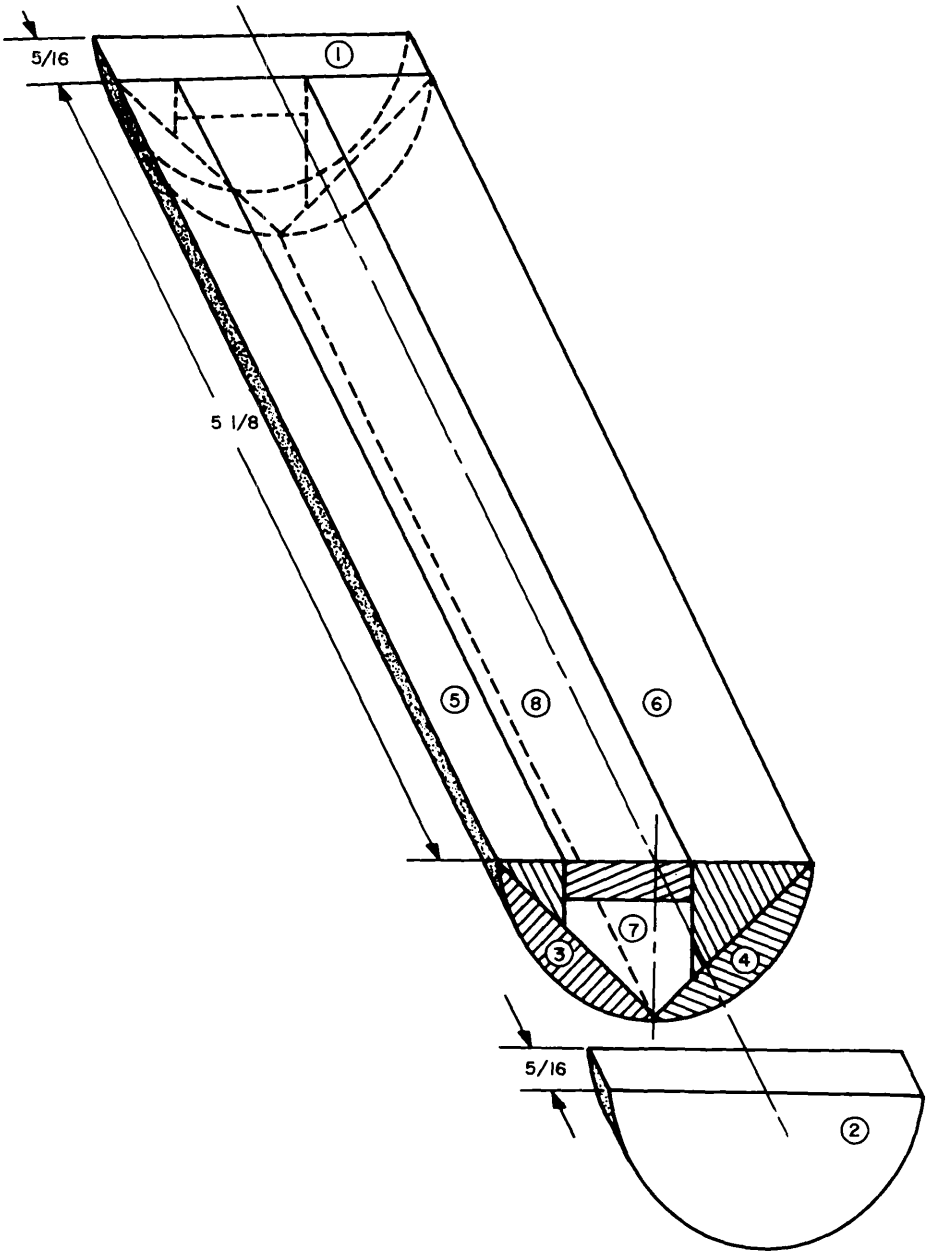

Fig. 33 - Relative segment location of sample 3

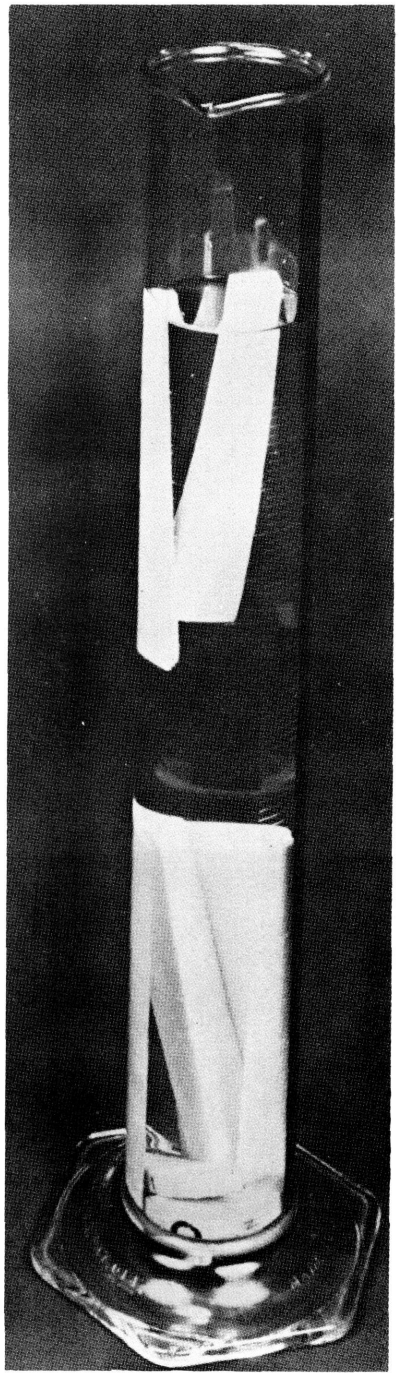

Fig. 34 - Outer segments of sample 3 sink; inner segments float

Table 6
Flotation Test for Segments of Sample 3 Under Pressurization for 1 Hour at 3000 psig

Segment Number*	Before Pressurization		After Pressurization		Percent Change	
	Weight (g)	Spec. Gravity (g/cc)	Weight (g)	Spec. Gravity (g/cc)	Δ Weight (%)	Δ Spec. Gravity (%)
1	3.48	0.765	5.25	1.103	50.9	44.2
2	3.49	0.740	5.20	1.075	49.0	45.3
3	9.17	0.792	13.31	1.108	45.2	39.9
4	9.41	0.741	13.13	1.057	39.5	42.7
5	4.01	0.776	5.13	0.932	27.9	20.1
6	7.01	0.800	10.79	1.157	53.9	44.6
7	10.80	0.786	15.97	1.130	47.9	43.8
8	8.29	0.677	9.70	0.811	17.0	19.8

*See Fig. 33 for segment identification.

Table 7
Syntactic Foam Life Expectancy

Sample Number	Exposure		Specific Gravity			Life Expectancy (yrs)
	Period (days)	Pressure (psi)	Original (g/cc)	After Exposure (g/cc)	Δ S.G. (g/cc)	
1*	1349	3,000	0.681	0.866	0.185	6.4
2	1413	10,000	0.765	1.183	0.418	2.72†
3	1160	10,000	0.713	1.047	0.334	2.75†
4	1002	10,000	0.709	0.918	0.209	3.8
5	1253	10,000	0.712	0.946	0.234	4.2
6‡	1056	10,000	0.726	0.840	0.114	7.0
7	1041	10,000	0.718	0.880	0.162	5.0
8	1056	10,000	0.726	0.797	0.071	11.2
9	1041	10,000	0.720	0.810	0.090	8.9
10§	296	5,000	0.611	0.630	0.019	16.6
11	296	5,000	0.602	0.623	0.021	15.4
12	299	10,000	0.687	0.710	0.023	11.2
13	299	10,000	0.684	0.707	0.023	11.3

*Set 1.
†Samples which sank.
‡Set 2.
§Set 3.

Appendix

THE USE OF SONIC ENERGY FOR THE MEASUREMENT OF SOME PHYSICAL PROPERTIES OF SYNTACTIC FOAM

It is possible, by judicious selection of sonic frequencies and the physical size of test specimens under investigation, to easily obtain certain physical properties concerning the specimen material. Physical properties obtainable by sonic means are those associated with the characteristic acoustic impedance ρc of the material.

The bulk modulus is of specific interest to the task group investigating the properties of syntactic foam. The bulk modulus can be obtained from Poisson's ratio and Young's modulus. For solids and liquids:

$$E = \rho c^2/g \ , \tag{A1}$$

where

E = Young's modulus (lb/ft^2),

ρ = density (lb/ft^2),

c = speed of sound (ft/sec),

g = gravitational constant (ft/sec^2), and

μ = Poisson's ratio.

If the test specimen were a liquid, E would be the bulk modulus. Since the shear and tensile stresses of a solid are not equal, it is necessary to obtain the value of Poisson's ratio or the modulus of shear or rigidity G before the bulk modulus of a solid can be obtained.

These three properties of a solid are related by

$$E = (1 + \mu)\ 2G \ . \tag{A2}$$

The shear modulus can be obtained directly through the performance of a tortional test or indirectly by acoustics. The bulk modulus is given by

$$K = E/3(1 - 2\mu) \ . \tag{A3}$$

From Eq. (A3) it can be seen that the bulk modulus can be obtained without knowing the shear modulus. In either case, however, we must obtain Poisson's ratio.

OBTAINING YOUNG'S MODULUS BY VIBRATIONAL VELOCITY

In this method Young's modulus is obtained by determining the velocity of the fundamental longitudinal (flectural) vibration of a long, thin rod. In the test which was conducted the syntactic foam rod was 18 in. long by 3/4 in. in diameter.

The rod was supported at its midlength only — so the ends were free to vibrate. With this support system only one nodal point exists for the fundamental frequency. The rod length is then equal to one-half the longitudinal wavelength of the fundamental frequency of the rod. The method used to vibrate the rod and to detect the fundamental frequency was as follows: A thin (5-mil) plate made of transformer-iron material was cemented to each end of the test rod. An electromagnetic transducer (pickup) was positioned at each end of the rod facing the metal plates. One pickup was used to generate the vibrational frequency; the second transducer acted as the pickup receiver. Energizing the generator pickup with a signal generator excites the rod in a longitudinal mode. This vibration modulates the magnetic field strength being sensed by the receiving pickup. The lowest frequency which develops the maximum signal amplitude in the receiver will be the fundamental frequency of the vibrating rod. With this frequency the velocity of the longitudinal vibration C_L and Young's modulus can be obtained:

$$E = 4f^2L^2\rho/g \ , \tag{A4}$$

where $C_L = 2fL$.

For convention this vibrational velocity will be referred to as the lower velocity. The upper, longitudinal, acoustic velocity is obtained by the use of ultrasonics.

DETERMINATION OF THE ACOUSTIC VELOCITY BY ULTRASONICS

The upper velocity, or the velocity of the longitudinal acoustic wave in syntactic foam, was determined by the more commonly used method of ultrasonics. In the ultrasonics method an instrument operating in the 500 Hz to 2 MHz region was found suitable for the syntactic foam which was tested. The Sonoray, a commercially available instrument, was used. Two models of this unit were available. One model used only one transducer as the projector and receiver. The second model used two transducers; one projector and one receiver. The operating principle of both models is the same. Where the first model measured the round-trip travel time, the second model measured the one-direction travel time only. Measurements were carried out on blocks that were 4 in. x 4 in. x 18 in. long. The acoustic ray was beamed along the 18-in. dimension.

The 4-in.-diam oscilloscope of the Sonoray gives a presentation of the transmitted and received pulses. The interval between the two "pips" is the travel-time lapse of the acoustic wave through the material being tested. The length of the test specimen divided by the indicated time interval is the speed of sound of the longitudinal wave. For Eq. (A5) this speed is denoted as the upper velocity C_u. Sonic velocities are always greater than the vibrational velocities.

By use of the lower and upper velocities, Poisson's ratio may be obtained without prior knowledge of the shear modulus by letting

$$C_L/C_u = K_1 \ .$$

Then

$$\mu = \left\{\left[(1 - K_1^2)^2 + 8(1 - k_1^2)\right]^{1/2} - (1 - k_1^2)\right\} 1/4 \ . \tag{A5}$$

The bulk modulus can now be obtained by substituting the value of μ and E into Eq. (A3).

As indicated by Eq. (A2) it is possible to determine the shear modulus by a technique similar to that for obtaining the upper velocity — in other words, by ultrasonics. However, pure shear waves are not as easy to generate as are the longitudinal ones. Nonetheless, with a little care and a little more acoustic power, shear waves can be realized. In this method Poisson's ratio is given by

$$\mu = (1 - 2K_2^2)/2(1 - K_2^2),$$

where

$$K_2 = C_s/C_L$$

in which C_s is the shear velocity, and C_L is the longitudinal velocity.

Describing a specimen through its acoustic impedance characteristics assumes that the specimen is, or behaves as, a completely homogeneous material. Whether syntactic foam can be considered a homogeneous material has not been sufficiently well established. Since the diameters of the hollow filler material (glass beads) and the ratio of beads to epoxy can be varied, it may be well to relate the physical properties of the syntactic foam to these variables.

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13. ABSTRACT

Syntactic foam samples were tested in fresh water under pressures of 3000, 5000, and 10,000 psi at 2°C and at room temperature (about 25°C). The samples were 1-3/4-in.-diam, 6-in.-long solid cylinders. The long-term hydrostatic test results, presented here, indicate that the rate of water absorption continues almost linearly with exposure time.

Based on the data presented at the present time the life expectancy (no longer buoyant) of a good grade of 42-lb/ft^3 syntactic foam at 10,000 psig is estimated to be about 10 years.

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