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STRENGTHS OF SULFUR-BASALT CONCRETES

UNITED STATES DEPARTMENT OF THE INTERIOR

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By Lester J. Crow and Robert C. Bates

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STRENGTHS OF SULFUR-BASALT CONCRETES

by

Lester J. Crow [1] and Robert C. Bates [1]

ABSTRACT

This study advances the use of elemental sulfur in structural materials by demonstrating its value in bonding high-strength, well-graded basalt aggregates to form sulfur-basalt concretes. Sulfur is an excellent bonding agent, and the strength of a thermoplastic sulfur-aggregate mixture depends, to a large degree, on the strength and grain-size distribution of the aggregate used. A 3-cubic-foot, electrically powered mixer with heat applied to the barrel was used to mix the sulfur and aggregate. Sulfur content of several mixtures was varied to obtain the best workability with a minimum of sulfur in excess of that necessary to fill the voids. The average grain sizes and grain-size distributions were also varied to determine their effects on strength. Unconfined compression tests of forty-five 6- by 12-inch cylinders yielded average strendths of 3,348 psi to 10,398 psi. The highest strength single cylinder was 10,717 psi. The cylinders were tested from 24 hours to 6 days after pouring; after 24 hours the rise in strength was very slight. From the results obtained it appears that sulfur can be a useful construction material.

An evaluation was made of a high-vacuum application for sulfur concrete. Sulfur has a high vapor pressure at elevated temperatures, 50° to 100° C; consequently, significant losses by sublimation can be expected. At lower temperatures the losses are not significant; however, this factor will limit its use at a higher temperature.

INTRODUCTION

The Bureau of Mines, sponsored by the National Aeronautics and Space Administration (NASA Contract No. R-09-040-001, April 1965), is studying various aspects of lunar material sampling and mineral exploitation. One phase of this was to advance the ground-support technology in the areas of both terrestrial and extraterrestrial mining. To accomplish this goal, research has been conducted on ground-support materials that have a potential for utilization in both areas. One such material is sulfur. Not only can sulfur provide an economical, effective solution to terrestrial construction and

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ground-support problems, it also might occur on the lunar surface and could, thereby, serve in an equivalent capacity for lunar-shelter construction.

The literature on the use of sulfur as a construction material points out sulfur's stability as a foam (3),[2] a high-strength aggregate bond (4), and a high-strength coating (2). Dale and Ludwig (4) at the Southwest Research Institute, San Antonio, Tex., performed the basic research on sulfur-aggregate concrete; however, as they point out, the mixture strengths were low (400 psi to 6,000 psi, median about 3,200 psi) because the limestone aggregates they used were generally of low strength. One mixture with a basalt aggregate had a compressive strength of 6,700 psi. It appears that the strength of a well-graded sulfur-aggregate concrete would then depend on the strength of the aggregate as suggested by the one mixture with basalt.

Essentially no work had been done using basalt, one of the rocks that appear to be indigenous to the lunar surface. Some forms of basalt yield a high-quality aggregate, and a strong aggregate when combined with sulfur could produce a top-quality construction material.

Laboratory experiments were conducted to measure the unconfined compressive strength of various sulfur-basalt concretes. This report describes the methods used and gives the results of these tests.

PROPERTIES OF SULFUR

Sulfur melts at 115° C and thickens to a dark amber liquid at 160° C. It has three allotropic forms: monoclinic, orthorhombic, and amorphous (5). Whether the form is monoclinic or orthorhombic depends on heat, curing temperature, and time. Sulfur, when it first congeals, is in the form of monoclinic crystals. On further cooling, it takes on the stabler orthorhombic form. The unconfined compressive strength is about 3,300 psi. Sulfur is an excellent bonding material, and it shrinks upon cooling.

The low melting point of sulfur poses one problem for its use in a lunar environment. Sulfur-supported structures could not be exposed during the lunar day, where the temperatures are estimated to range as high as 120° C ± 50°. However, the use of sulfur underground may be feasible, because the overlying lunar materials will provide an adequate heat barrier.

Another problem is the tendency for sulfur to sublime well below its melting point because of its relatively high vapor pressure (4.7×10^{-5} torr at 50° C; 6.6×10^{-3} torr at 100° C). Following the method of Jaffe and Rittenhouse (8), the sublimation rate can be calculated from the equation

$$S = 1.85 \times 10^{6} \frac{P}{\rho}\sqrt{\frac{M}{T}}, \qquad (1)$$

where S = rate of sublimation in cm/yr,

P = vapor pressure in torr (mm Hg),

[2]Underlined numbers in parentheses refer to items in the list of references at the end of this report.

ρ = density of solid, g/cm^3 (2.07 g/cm^3 for sulfur),

M = molecular weight of vapor (8 times atomic wt of sulfur),[3]

and T = temperature in degrees Kelvin.

The derivation of this equation assumes that none of the molecules leaving the surface returns due to collision with atmospheric molecules, a reasonable assumption in lunar vacuum.

Applying equation 1 and using the available information on sulfur vapor pressures shows the dependence of sublimation rate on temperature. At 60° C (140° F) a sulfur layer over 3-1/2 feet thick would be lost by sublimation in a year. Near room temperature, 20° C (68° F), only 1/3 inch per year would be lost by sublimation. For a lower temperature such as -20° C (-4° F), that might exist just below the lunar surface, the sublimation rate is a microscopic 0.0007 inch per year. Table 1 and figure 1 show sublimation rates calculated for a wide range of temperatures. The data given in table 1 and figure 1 must be viewed with a little caution because the literature on sulfur vapor pressures is not as reliable as it should be. Fouretier (7) appears to present the best data, as they were based on a series of experimental determinations in the temperature range 20° to 80° C. His data were described by the equation

$$\log P = 11.664 - \frac{5.166}{T}, \qquad (2)$$

where P = vapor pressure in torr and T = temperature in degrees Kelvin.

Equation 2 was used to calculate the vapor pressures given in table 1. The extrapolation from +20° C to -50° C is certainly open to criticism, since there are no experimental data below 20° C available. Furthermore, the vapor pressures reported by different experimenters vary significantly, suggesting that the calculated vapor pressure and sublimation rates might be in error by a factor of two from the actual but unknown case.

It is obvious from these data that steps will have to be taken to shield the material from high temperatures and from the lunar vacuum, or considerable quantities of it will be lost. These safeguards apply both in handling and after emplacement.

[3]Sulfur vaporizes as S_8 molecules.

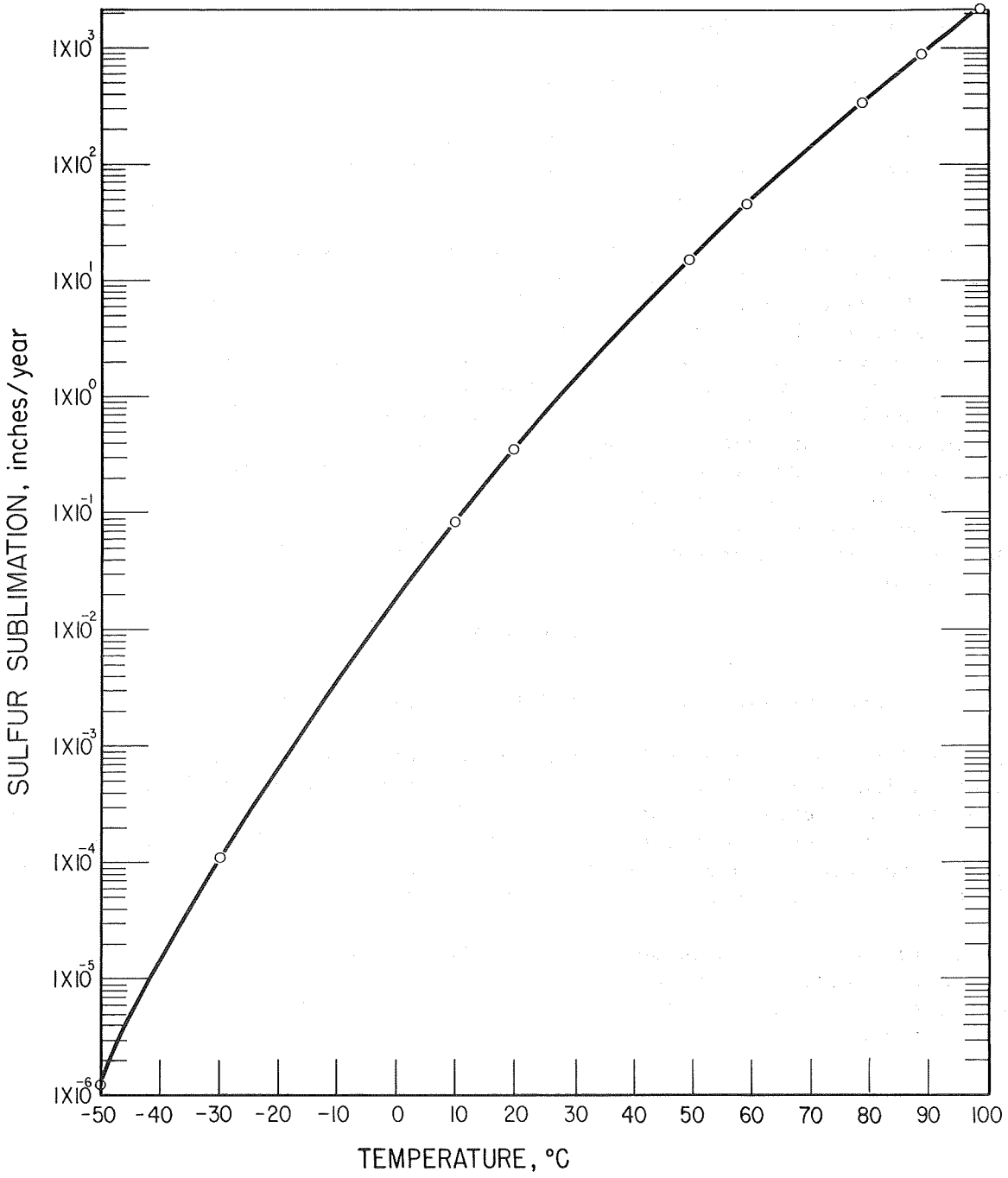

FIGURE 1. - Plot of Sulfur Sublimation Rates As a Function of Temperature.

TABLE 1. - *Calculated sublimation rates of sulfur in vacuum for various temperatures*

Temperature, ° C	Vapor pressure, torr	Sublimation rate	
		Cm/yr	In/yr
100	6.6×10^{-3}	4.9×10^{3}	1.9×10^{3}
90	2.7×10^{-3}	2.1×10^{3}	8.1×10^{2}
80	1.1×10^{-3}	8.2×10^{2}	3.2×10^{2}
70	4.0×10^{-4}	3.1×10^{2}	1.2×10^{2}
60	1.4×10^{-4}	1.1×10^{2}	4.4×10^{1}
50	4.7×10^{-5}	3.7×10^{1}	1.4×10^{1}
40	1.5×10^{-5}	1.1×10^{1}	4.6×10^{0}
30	4.2×10^{-6}	3.4×10^{0}	1.3×10^{0}
20	1.1×10^{-6}	9.1×10^{-1}	3.6×10^{-1}
10	2.6×10^{-7}	2.2×10^{-1}	8.7×10^{-2}
0	5.6×10^{-8}	4.8×10^{-2}	1.9×10^{-2}
-10	1.1×10^{-8}	9.4×10^{-3}	3.7×10^{-3}
-20	1.8×10^{-9}	1.6×10^{-3}	6.3×10^{-4}
-30	2.6×10^{-10}	2.4×10^{-4}	9.4×10^{-5}
-40	3.2×10^{-11}	3.0×10^{-5}	1.2×10^{-5}
-50	3.2×10^{-12}	3.1×10^{-6}	1.2×10^{-6}

LABORATORY EXPERIMENTS

Work by Dale and Ludwig (4) supplied some background on which to organize the experiments. Their work indicated that samples in which all of the available void space was filled with sulfur were stronger than those in which the aggregate was only coated by sulfur. A fairly well-documented sulfur-aggregate curing curve for 4.5- by 9.0-inch cylinders was given in their report. Figure 10 in their report shows that 60 percent of the 28-day compressive strength was achieved in 45 minutes after pouring. In 3 hours the strength was 81 percent of the 28-day strength. In 12 hours it was 87 percent, and in 24 hours it was 88 percent. Beyond this time there was a very gradual increase in strength to the 28-day strength. Since there are no time and moisture content reactions taking place in a sulfur concrete as there are in a regular concrete, the major criterion for curing is the speed of heat removal. As mentioned earlier, it appeared that the strength of the aggregate also affected the strength of the sulfur concrete. Less obvious than these factors was a clue to the influence of grain size on the strength of sulfur concrete. After examining, recalculating, and plotting Dale and Ludwig's data with strength as a function of two grain-size distribution parameters, there appeared to be a trend toward higher strengths as the average grain size[4] became smaller and the coefficient of uniformity[5] became larger. Hence, it

[4]Average grain size (50 percent finer grain size) is the particle size, usually in millimeters, below which is half of the sample by weight. It is usually taken from a grain-size distribution graph.

[5]Coefficient of uniformity (D_{60}/D_{10}) is the ratio of the 60-percent finer grain size divided by the 10-percent finer grain size, and describes the particle-size distribution in a sample. A coefficient of uniformity of 1 indicates a sample consisting of only one size of particles (uniform).

appeared for the purpose of this experiment that the grain-size parameters, among other factors, had to be either controlled or measured.

Consideration of all of the information available, along with some deductive reasoning, led to the belief that strength as the dependent variable would be some nonlinear function of the independent variables. The average grain size and the coefficient of uniformity were chosen as the independent variables to be studied, and a slight excess of sulfur was specified for most of the tests to insure filling all the void space. A fine-grained basalt aggregate was chosen because of its high strength and availability. Standard 6- by 12-inch cylinders were chosen for all unconfined compression tests.

Once some starting points were established, the next consideration was the planning of the experiment to answer the salient questions and to supply the answers with a satisfactory statistical base. The type of experimental designs involving fixed increments of the independent variable, such as a factorial design, did not appear applicable here because of the time and expense required to properly composite the aggregates. It would have been necessary to acquire and screen several tons of aggregate, and there was no assurance of properly bracketing any maximum or minimum strength values. Likewise, it was felt that it would not be economical to sample a large number of combinations in a measured but uncontrolled fashion and then employ regression-analysis techniques to find the maximum and minimum values.

After eliminating these and several other possible statistical experimental designs, it appeared that the most suitable technique was the Method of Steepest Ascent (9). In this method tests are made using three or four combinations of the independent variables at some convenient arrangement about a starting point. The first results are analyzed to determine how the magnitude of the independent variables should be changed to achieve the fastest improvement in the result, in this case higher strength. Then a test (or two) is run at some convenient level away from the starting group in the direction indicated by the first analysis. These new results are examined along with the preceding results to determine what change, if any, should be made in the direction of the investigation. This method is most easily visualized by considering the analogy of a mountain climber who adjusts his east-west position and his north-south position in such a manner that he climbs the mountain by the steepest path possible. As in the analogy, eventually the top is reached, and then all that remains is to establish the size and shape of the peak. Where applicable, this method can achieve the desired result faster and at less expense than the more formal designs. The main criterion is that new data must be analyzed immediately to determine the next step.

The materials, equipment, and procedures used in this study are described in the following sections.

Materials

Bright elemental sulfur with a minimum purity of 99.9 percent was used. The solid flake form was purchased in 50-pound bags and in this form the sulfur had a unit weight of 69 pounds per cubic foot. The flake form was used because it was more convenient in weighing and easier to mix than the lump form.

The aggregate was a crushed-flow basalt rock derived near Spokane, Wash., from the northeastern limit of the Columbia River Basalt formation. This local material is representative of the flow basalt selected by Fogelson (6) in the Columbia River Basalt at Madras, Oreg., as a simulated lunar basalt. Both rock sites have the characteristic columnar flow basalt structure in situ. Petrographically, both rock materials exhibit dense, fine-grained mineral properties with no vesicular structure. The compressive and tensile strength properties of the Spokane flow basalt are also believed to be similar to those of the simulated lunar flow basalt at Madras, Oreg., site (6). Two sizes of aggregate that were produced by a standard crushing and screening plant were chosen for these experiments. The coarser was minus 1-1/4 inches and conforms to a No. 57 graded aggregate (1, p. 19); it is represented by grain-size distribution curve *1* in figure 2. The finer aggregate was produced as minus 5/8 inch; however, for these tests the plus 4-mesh portion was removed, leaving an aggregate with the grain-size distribution given by curve *2* in figure 2. One consideration in the choice of commercially produced aggregates was that any beneficial results from the experiments could be easily scaled up without the necessity of producing specially composited grain-size distributions. Any grain distribution between the coarse and fine aggregates, like curve *3* in figure 2, was produced by mixing appropriate amounts of the two.

Commercially prepared 6,000- and 10,000-psi concrete-cylinder capping compounds were used to cap the test cylinders prior to compression testing. Standard capping procedures were used.

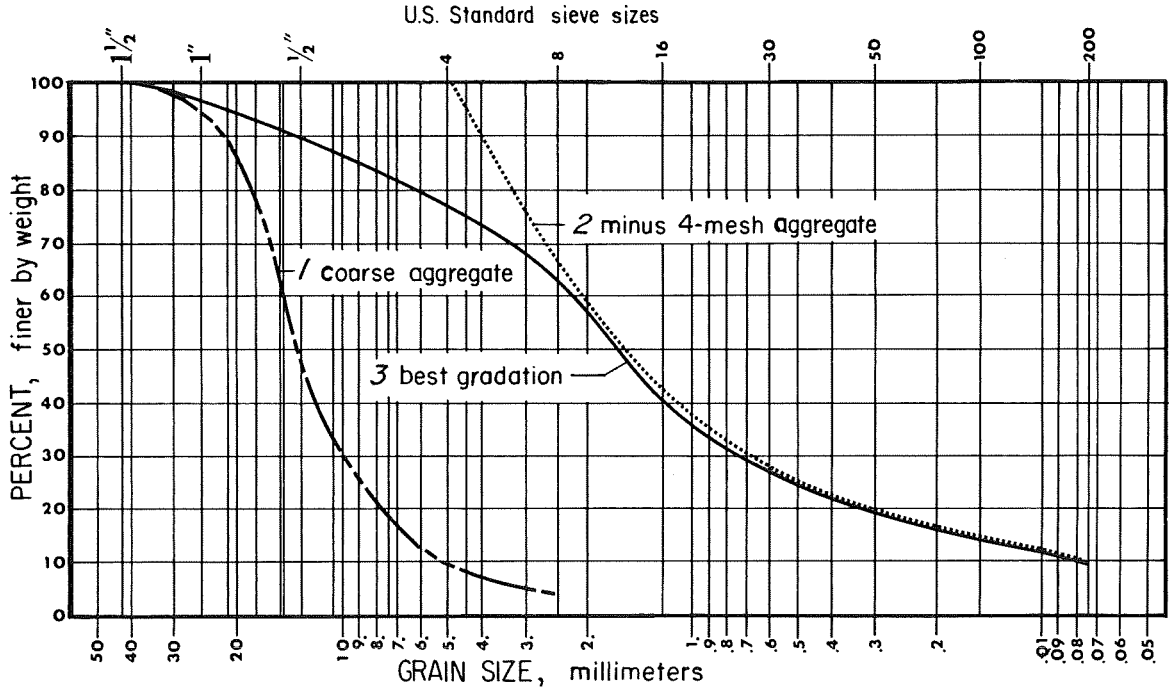

FIGURE 2. - Grain-Size Distribution Curves of Three Aggregates Used in Studies

Equipment and Procedure

A 3-cubic-foot, electrically powered concrete mixer was used to mix the aggregate. To obtain better mixing, the flights in the mixer were extended from the bottom to the top of the barrel. The end of the mixer barrel was covered with a cap to prevent excessive loss of fines and to conserve the heat in the barrel while the aggregate was being heated. It also tended to reduce the possibility of sulfur ignition, in case portions of the mixer barrel became too hot. A metal funnel was attached to the mixer frame, and two infrared lights mounted underneath on the mixer frame were used to keep the funnel warm when the mixer was in use.

The mixer barrel and its contents were heated by two burner heads using butane gas. The burner heads, flared at the ends to allow for more coverage of the barrel surface, were mounted from the bottom of the mixer on the opposite side from the funnel and positioned 6 inches from the barrel. The butane tank was equipped with a pressure gage, and at 10 pounds of gas pressure, the

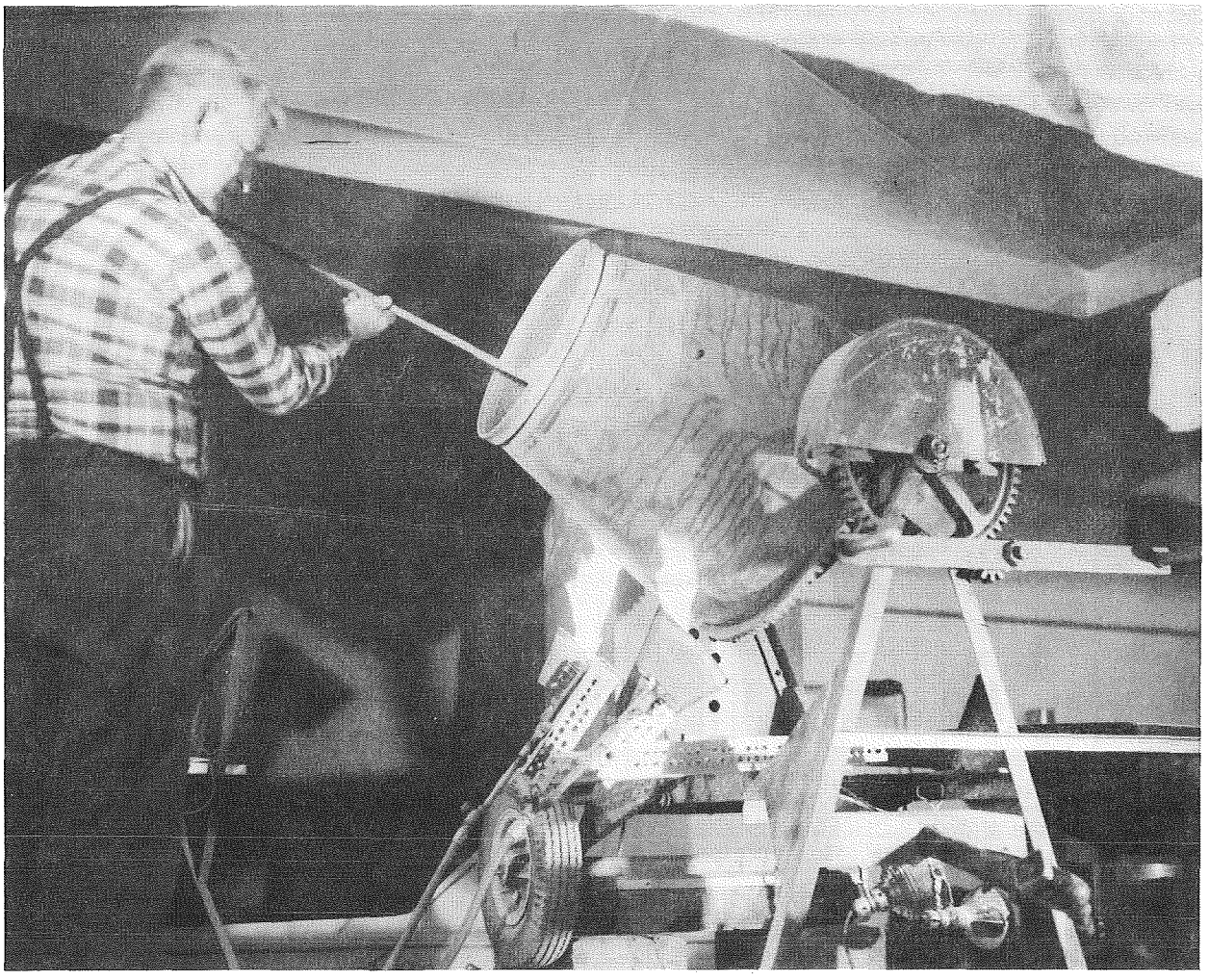

FIGURE 3. - Measuring Temperature of Mix With Thermocouple Probe.

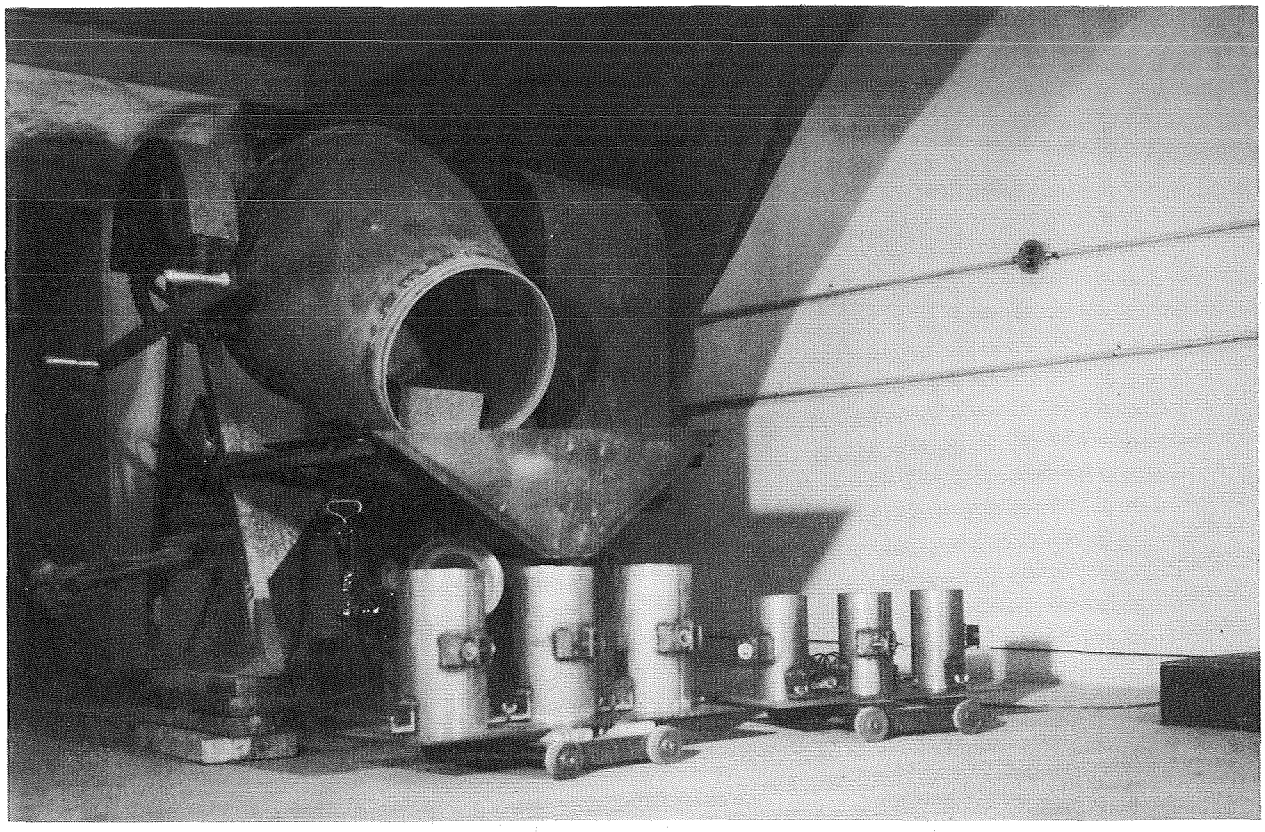

FIGURE 4. - Steel Molds (6- by 12-Inch) Used in Making Compression Test Cylinders (Left) and Smaller Molds Used in Incidental Tests (Right).

burner heads deliver approximately 55,200 Btu per hour. A thermocouple in a perforated steel rod (fig. 3) was used to check the temperature of the aggregate.

Steel molds, 6-inch-diameter by 12 inches high (fig. 4, left) were used to cast the test cylinders. The smaller molds (fig. 4, right) were used for several incidental tests. They were too small for casting specimens with large-size aggregate; therefore, they were not used in the main series of compression tests. The molds were preheated on a large hotplate to prevent premature cooling of the mix when the test specimens were being cast. Small dollies were used to move them around after being removed from the hotplate. Test cylinders (fig. 5), cast in the 6- by 12-inch molds, level full, weighed an average of 32 pounds.

The first cylinders were broken using a 200,000-pound-capacity Tinius Olsen[6] compression tester. On later test cylinders, through better selection of grain sizes and improved techniques, the unconfined compressive strengths of the test specimens exceeded the capacity of this machine. Rather than try

[6]Reference to specific trade names is made for identification only and does not imply endorsement by the Bureau of Mines.

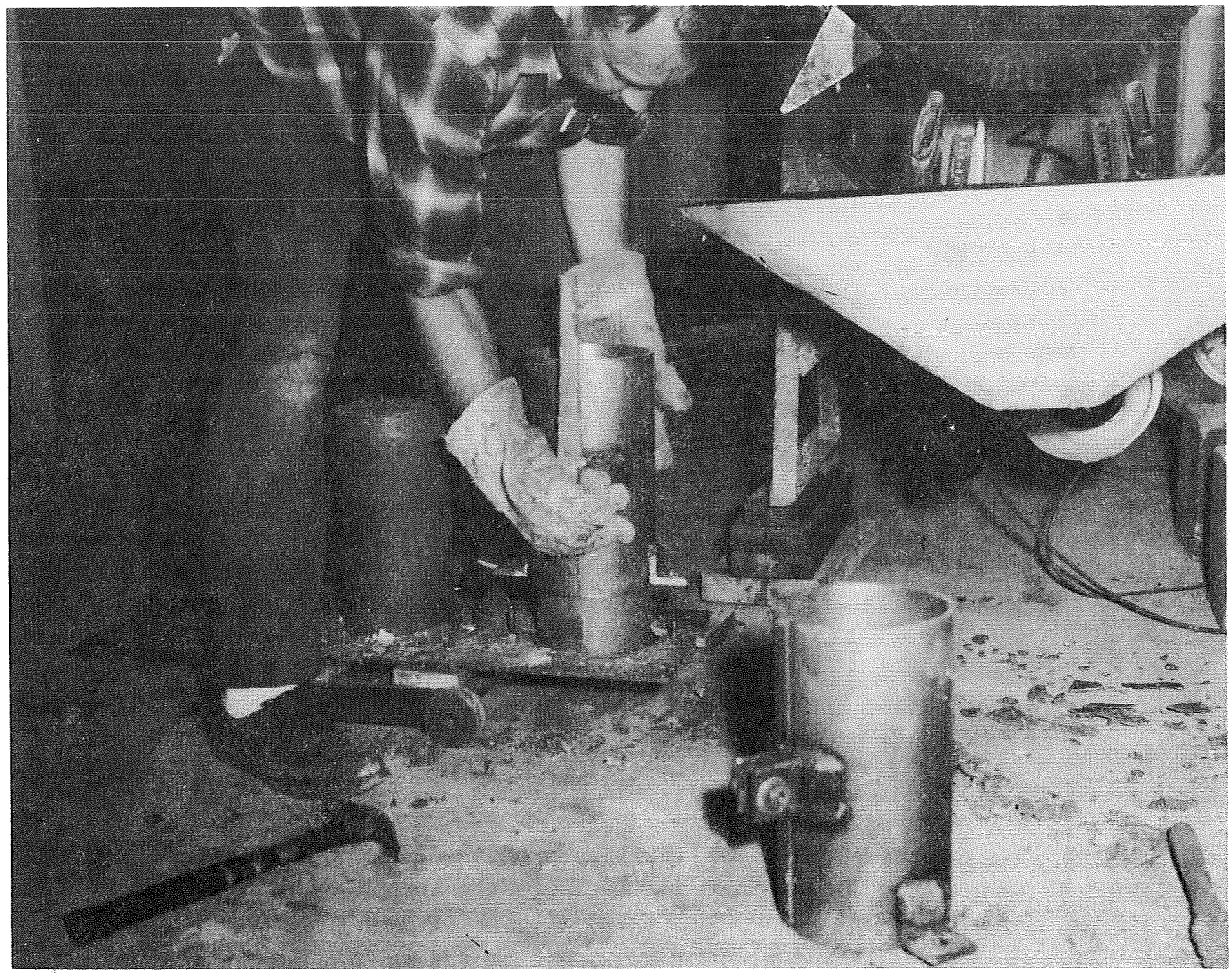

FIGURE 5. - Compression Test Cylinders (6- by 12-Inch) Being Removed From Mold.

to use the smaller test cylinders, the improved specimens were broken on a 400,000-pound Tinius Olsen universal testing machine. On both machines a compression loading rate of about 1,500 psi per minute was used.

Workability

Workability is the quality or state of being workable. The amount of sulfur used in the aggregate mixes has to be carefully controlled to completely fill the voids and yet maintain good workability. The index of workability is described in Dale's report (4):

> The sulfur-sand-aggregate mixture should be of such a character that when the mix is poured onto a level surface from the height of 18 inches, the height-to-diameter ratio of the resultant poured material should be between 1:6 and 1:8 and there should be no trace of excess sulfur, which can be easily distinguished by its show of color or by its crystalline patterns that develop in solidifications.

Insufficient sulfur leaves voids in the specimen that reduce the strength considerably, and too much sulfur causes excess shrinkage, that also reduces the strength. When the proper mix is attained, the whole mass moves easily upon being heated to the proper temperature, and there is no free sulfur showing.

Heating

The aggregate was first heated in the mixer to a temperature between 140° and 150° C. This working temperature range was chosen because it is considerably above the melting point of sulfur and below the temperature, 160° C, where liquid sulfur becomes viscous. After achieving this aggregate temperature range, the sulfur was placed on the hot aggregate and allowed to melt and mix without any further addition of heat. The mix temperature dropped somewhat during this time, but not enough to cause problems in working the mixture in the molds before it congealed.

A low heat was found to be most desirable in bringing the aggregate to the proper temperature. When a high heat was applied, the mixer barrel became considerably hotter than the aggregate contents; sometimes it was well above the ignition point of sulfur. In such cases tiny fires would start when the sulfur was introduced into the mixer, but these were quickly extinguished by the continued mixing and covering of the mixer barrel. During the heating of the aggregate, the temperature was always measured directly with a thermocouple probe. However, with a little experience in checking temperatures at given time intervals on a selected weight of aggregate, an aggregate charge could be brought to the proper temperature on a time basis alone.

Pouring

The mixer is kept revolving during a pour. By keeping the funnel and molds heated, the workability of the poured material was maintained for about 60 seconds. The molds were filled in three lifts, with rodding between lifts to eliminate any large bubbles from entrained air and to produce some compaction. The molds were puddled and struck off as level as possible.

When the sulfur aggregate starts to cool in the mold, the top portion which is exposed to the air seals over almost immediately. If this crust is allowed to remain until the cylinder is completely cool, a flat pocket forms about an inch under the top surface and weakens the specimen. This void was eliminated by breaking the crust and continuing to puddle the material as it cooled. This eliminates the large void, but not necessarily all the medium-sized voids. To eliminate all large and medium-sized voids, the crust on top was broken, and some extra mix added with puddling continued until the specimen congealed. This procedure to eliminate voids is easily reproduced.

A similar problem with shrinkage would be encountered if the material were poured in larger molds. Since shrinkage affects the uniformity and density of any given pour and in turn reduces its ultimate strength, procedures would be needed to eliminate it by puddling the material as it cools and by adding a small amount of mix.

Results of Testing

A variety of aggregate-sulfur mixtures were examined in this series of tests. The average grain size, grain-size distribution, and percentage of sulfur were varied from test to test in a controlled manner to arrive at the highest strength sulfur concrete possible. Described in the following paragraphs are the results of the experiments. A few sulfur concretes were prepared, using sulfur and a coarse (minus 1-1/4-inch, plus 4-mesh) No. 57 graded aggregate, and a few were made with all fine (minus 4-mesh) aggregate. However, most of the sulfur concretes were made using a double-graded aggregate mixture consisting of coarse (No. 57) and fine (minus 4-mesh) aggregates. The complete gradations for each mix are given in table 2, and test data for each cylinder specimen are given in table 3.

TABLE 2. - *Sieve analysis of aggregates employed*

Sample	Amounts passing each laboratory sieve (square openings[1]), weight-percent										
	1-1/2-inch	1-inch	1/2-inch	Number 4	Number 8	Number 10	Number 16	Number 30	Number 50	Number 100	Number 200
A	100	95.00	48.00	9.5	4.00	-	-	-	-	-	-
1	100	99.16	89.94	61.91	41.46	-	28.08	-	16.07	12.10	8.64
2	-	100.00	98.87	77.80	52.34	-	35.67	26.63	19.56	14.51	10.50
3	-	100.00	82.68	68.04	50.32	-	34.50	25.22	18.43	13.43	9.42
4	-	100.00	92.12	78.56	55.00	-	38.24	-	22.35	16.77	12.10
5	-	100.00	95.10	86.54	53.74	-	34.64	-	19.68	14.94	11.02
6	100	96.88	86.93	83.07	58.95	-	41.92	32.86	17.49	12.01	7.77
7	-	-	-	100.00	66.44	-	38.06	27.26	19.86	14.41	10.05
8	-	-	-	100.00	58.91	-	31.89	19.84	12.27	6.94	3.79
9	-	-	-	-	-	100	72.22	48.76	32.49	21.18	12.00
10	-	-	-	-	-	100	76.97	55.23	39.47	27.97	19.01
11	100	90.20	75.00	51.50	36.00	-	23.80	15.0	10.40	9.20	8.00

[1]In inches, except where otherwise indicated. Numbered sieves are those of United States standard sieve sizes.

TABLE 3. - *Test data on individual specimens*

Sample	Aggregate, weight-percent		Sulfur, weight-percent	Average grain size, mm	Coefficient of uniformity	Failure load of individual cylinders, lb	Compressive strength		Standard deviation
	Minus 1-1/4-inch plus 4 mesh	Minus 4 mesh					Per cylinder, psi	Average, psi	
A	-	-	6.0	-	-	707	250	243	10
						667	236		
						([1])	([1])		
1a	37.2	37.1	25.7	3.30	51.1	64,000	2,264	3,348	1,553
						75,000	2,653		
						145,000	5,128		
1b	35.5	35.5	29.0	3.30	51.1	94,000	3,325	3,714	550
						116,500	4,103		
						([1])	([1])		
1c	39.0	39.0	22.0	3.30	51.1	130,000	4,598	5,022	374
						146,000	5,164		
						150,000	5,305		
1d	38.7	38.7	22.6	3.30	51.1	187,000	6,614	6,378	409
						187,000	6,614		
						167,000	5,906		

See footnotes at end of table.

TABLE 3. - *Test data on individual specimens*--Continued

Sample	Aggregate, weight-percent		Sulfur, weight-percent	Average grain size, mm	Coefficient of uniformity	Failure load of individual cylinders, lb	Compressive strength		Standard deviation
	Minus 1-1/4-inch plus 4 mesh	Minus 4 mesh					Per cylinder, psi	Average, psi	
2	34.6	43.3	22.1	2.20	47.5	(1)	(1)	7,171	738
						217,500	7,692		
						188,000	6,649		
3	30.3	47.6	22.1	2.30	40.7	(1)	(1)	8,029	701
						213,000	7,533		
						241,000	8,524		
4	26.0	52.3	21.7	2.01	50.0	234,500	8,294	8,276	25
						235,500	8,258		
						(1)	(1)		
5	15.4	61.5	23.1	2.10	46.7	267,500	9,461	8,695	663
						235,000	8,312		
						235,000	8,312		
6	8.7	69.6	21.7	1.70	22.7	303,000	10,717	10,351	372
						293,000	10,363		
						282,000	9,974		
7a	-	77.6	22.4	1.64	27.7	(1)	(1)	[2]10,398	-
						(1)	(1)		
						294,000	10,398		
7b	-	75.0	25.0	1.64	27.7	249,000	8,807	[2]9,196	341
						267,000	9,443		
						264,000	9,337		
7c	-	76.3	23.7	1.64	27.7	239,500	8,471	[2]8,686	189
						252,500	8,930		
						244,750	8,656		
7d	-	77.6	22.4	1.64	27.7	(1)	(1)	[2]9,584	450
						262,000	9,266		
						280,000	9,903		
8	-	[3]76.3	23.7	1.94	10.4	176,000	6,225	6,355	944
						155,000	5,482		
						208,000	7,357		
9	-	[4]75.0	25.0	.60	14.0	215,000	7,604	7,911	266
						228,000	8,064		
						228,000	8,064		
10	-	[4]76.3	23.7	.48	28.4	185,000	6,543	7,536	1,774
						183,250	6,481		
						271,000	9,585		
11	37.9	40.4	21.7	4.40	26.0	192,750	6,817	6,974	142
						(1)	(1)		
						201,500	7,127		
						(1)	(1)		
						195,000	6,897		
						199,500	7,056		

[1]Cylinder unacceptable for testing.
[2]Average of samples 7a-7d: 9,246 psi.
[3]Minus 4 plus 200 mesh.
[4]Minus 10 mesh.

A preliminary test using coarse, No. 57 graded aggregate and about 6 weight-percent sulfur (sample A) was made to examine the characteristics of a sulfur-deficient concrete. When the sulfur was melted over the coarse aggregate, only a thin coating of sulfur was retained on the particles, and any sulfur in excess of this thin coat ran like water through the aggregate to the bottom of the mixer. When poured, the free sulfur ran out first and collected on the bottom of the mold, leaving the remainder of the specimen an open cellular structure (fig. 6). Unconfined compression strengths of these

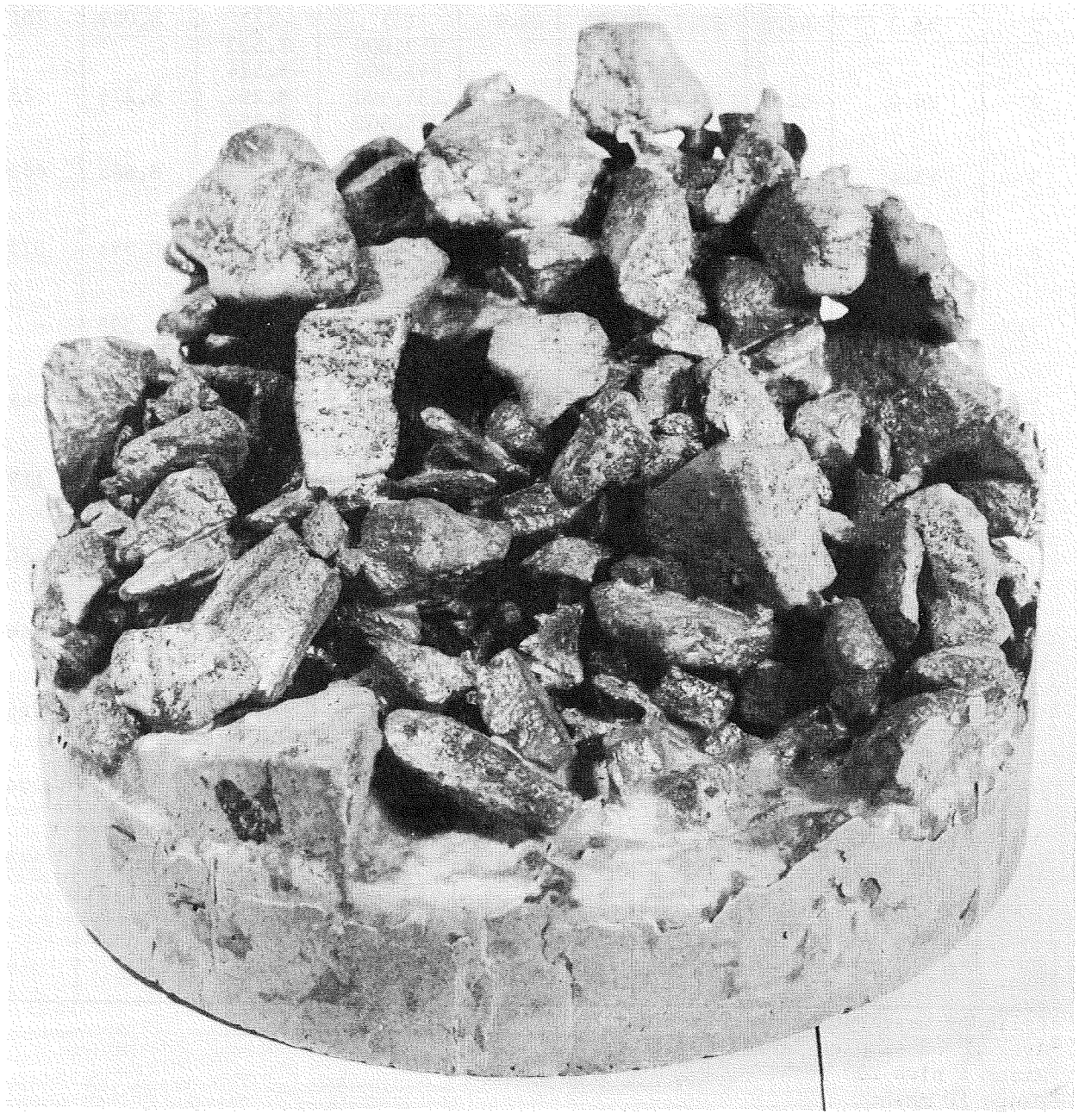

FIGURE 6. - Wetted and Partially Filled Coarse Aggregate.

specimens did not exceed 250 psi and were not good samples, since they were partially filled and partially open structures. It was quite evident by inspection that fine aggregate should be added to fill the voids in the coarse aggregate and hold the sulfur in suspension.

The first experiments using composited aggregates (sample 1) were made with equal portions by weight of coarse and fine material. In part, these experiments were designed to gain knowledge of the workability with different percentages of sulfur as well as to maximize the compressive strength. The first set of three cylinders (sample 1a) contained 25.7 weight-percent sulfur. The workability of this mix was good, but there was some free sulfur showing. These cylinders when tested broke in the top one-third of their height. Average unconfined compressive strength of these cylinders was 3,348 psi. A second set of three cylinders (sample 1b) contained 29 weight-percent sulfur. This mixture was quite fluid. While capping these cylinders, the top snapped off one of them, showing a large flat shrinkage void (fig. 7). Of the remaining two cylinders, the average unconfined compressive strength was 3,714 psi. On a third set of cylinders (sample 1c) 22 weight-percent sulfur was added. The workability of this material was good, and the average unconfined compressive strength was 5,022 psi, considerably higher than that in the

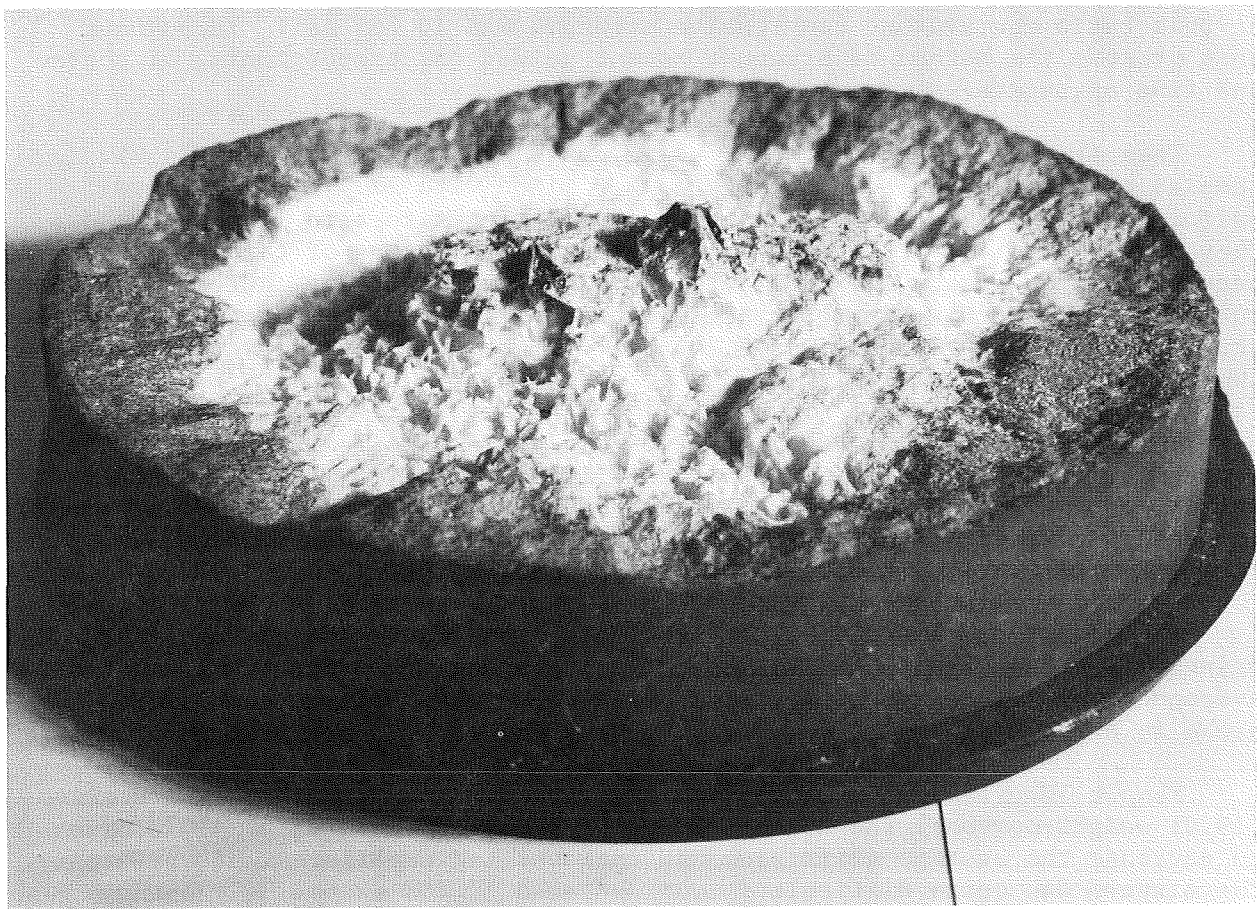

FIGURE 7. - Void Created by Shrinkage and Resulting Growth of the Sulfur Crystals.

prior tests. However, all of the cylinder failures were in the top one-third of the specimens. Inspection of the broken material revealed a flat void in the upper portion of each of the cylinders, but not as pronounced as the one shown in figure 7.

In seeking a way to eliminate voids, especially the flat one in the top center core of the specimen, another set of three cylinders (sample 1d) was made up of the same composition. When the specimen first sealed over on the top while cooling in the mold, the crust was broken open, and some of the hot sulfur aggregate mixture was added and puddled until the entire mix began to congeal. The average compressive strength of these specimens was 6,378 psi, 27 percent more than that of the previous ones. These samples broke in a classic double-cone fashion. On inspecting the broken pieces from these specimens, it was observed that more fine aggregate would be beneficial in more completely filling the void space between the coarse particles.

Analysis of the unconfined compression strengths indicated further improvement by decreasing the average grain size. The percentage of fine aggregate was increased and the strength increased. This process was continued (samples 2-6), and the breaking strengths were increased by adding fines until the fine-to-coarse aggregate ratio was 8-to-1 by weight (sample 6). At this ratio the average compressive strength was 10,351 psi. Throughout this process the Method of Steepest Ascent guided the proportioning of coarse and fine aggregate.

Then it was decided to use an all minus 4-mesh aggregate (sample 7). Samples of all minus 4-mesh aggregate when mixed with 22.4 weight-percent sulfur (sample 7a) became quite spongy and difficult to tamp into the molds. They were also a little erratic on the breaks. Four sets of three cylinders each were mixed with sulfur contents up to 25 weight-percent, but this did not substantially improve the workability of the mixture. The average compressive strength on these cylinders was 9,246 psi. Figure 8 shows the manner in which these cylinders broke.

In sample 8, most of the minus 200-mesh grain size was removed from the aggregate, giving the sample a lower coefficient of uniformity. The small amount, 3.8 percent by weight, of minus 200-mesh aggregate left in this sample caused the surface of the cylinder to look rough as the sulfur drained away. These samples averaged 6,355 psi compressive strength.

In another sample, No. 9, the average grain size was reduced further by using only minus 10-mesh material for the aggregate. These cylinders had an average compressive strength of 7,911 psi. Specimens of this composition broke in a blocky manner (fig. 9).

Sample 10 cylinders also used a minus 10-mesh gradation, but had double (19.01 weight-percent) the normal amount of minus 200-mesh material in order to decrease the average grain size and increase the coefficient of uniformity. The mixture was quite difficult to work because of its exceedingly spongy nature. The average compressive strength was 7,536 psi.

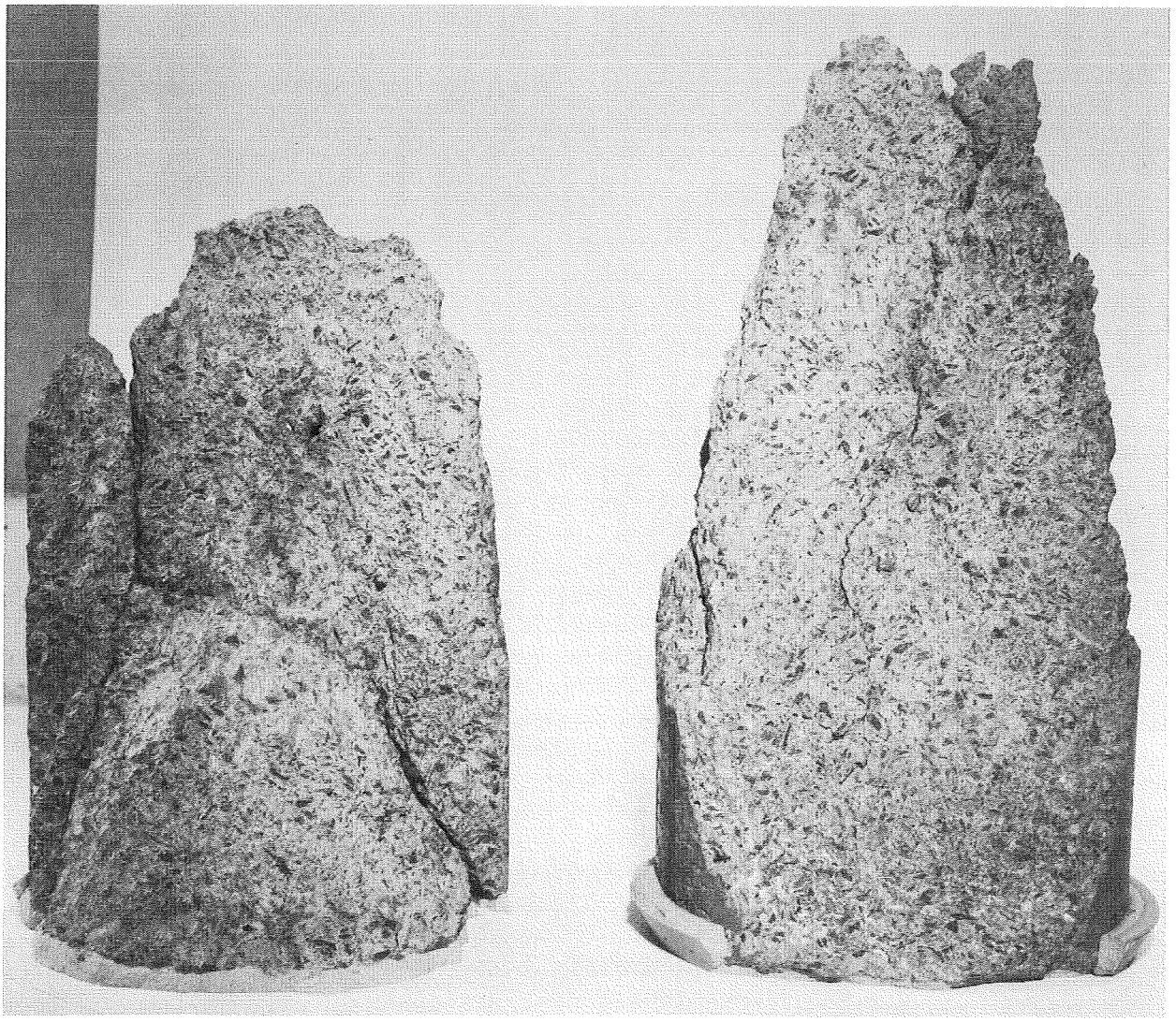

FIGURE 8. - Test Cylinder Using Minus 4-Mesh Basalt, Failed at 9,200 psi.

The last sample, No. 11, was manufactured to have a large average grain size and a coefficient of uniformity of 26.0. The average compressive strength was 6,974 psi.

The single sample that had the highest unconfined compressive strength of 10,717 psi was one containing 8-to-1 ratio by weight of coarse-to-fine aggregates with 22.7 weight-percent sulfur (sample 6). Figure 10 shows how this cylinder broke.

These results are summarized in table 4. One method of displaying these data is to contour the unconfined compressive strengths, as in figure 11. Here the abscissa and ordinate are the average grain size and coefficient of uniformity, respectively. The contour lines indicate the unconfined compressive strength as a function of both grain-size variables. The grain sizes for

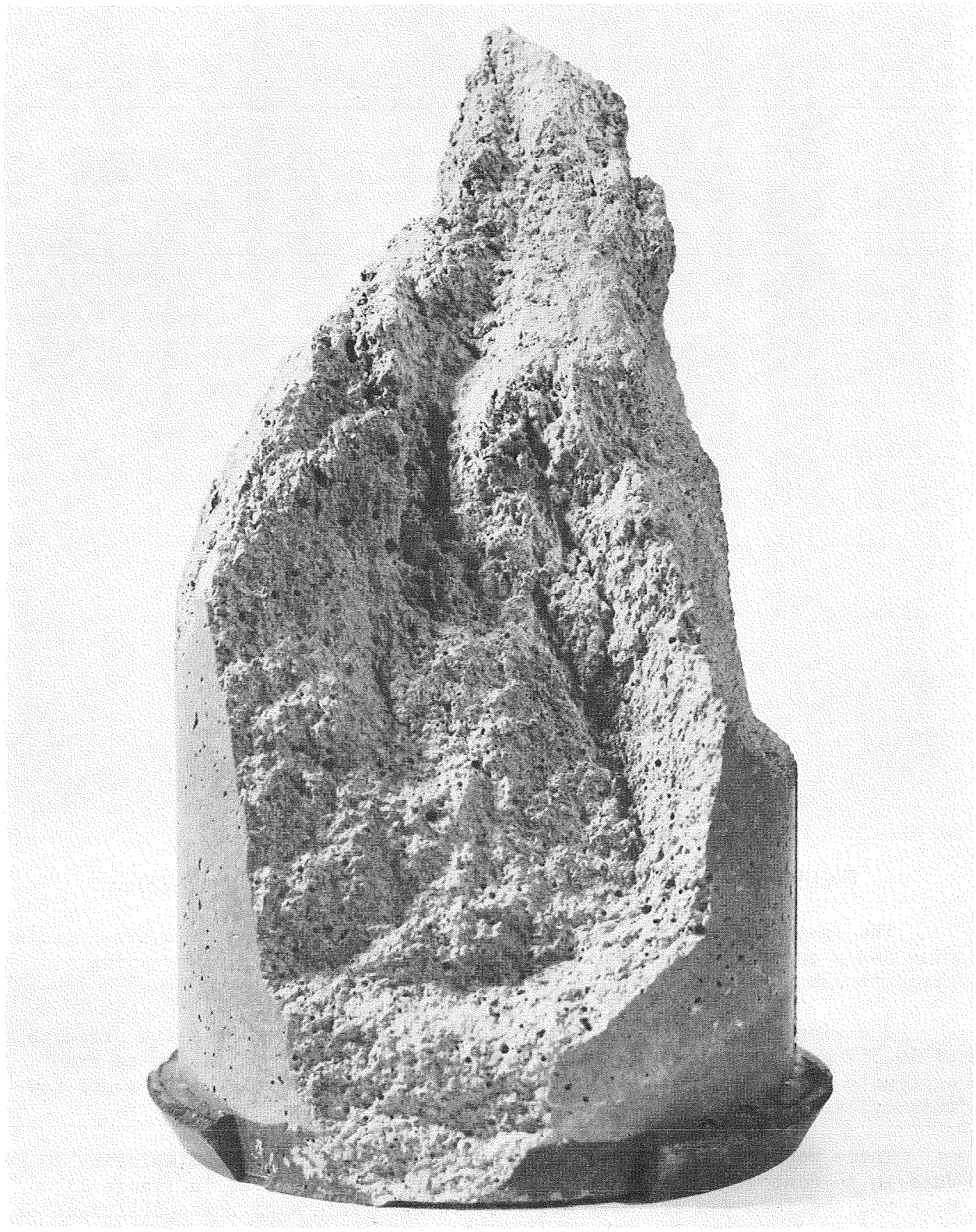

FIGURE 9. - Test Cylinder Using Excess Fines, Failed at 7,900 psi.

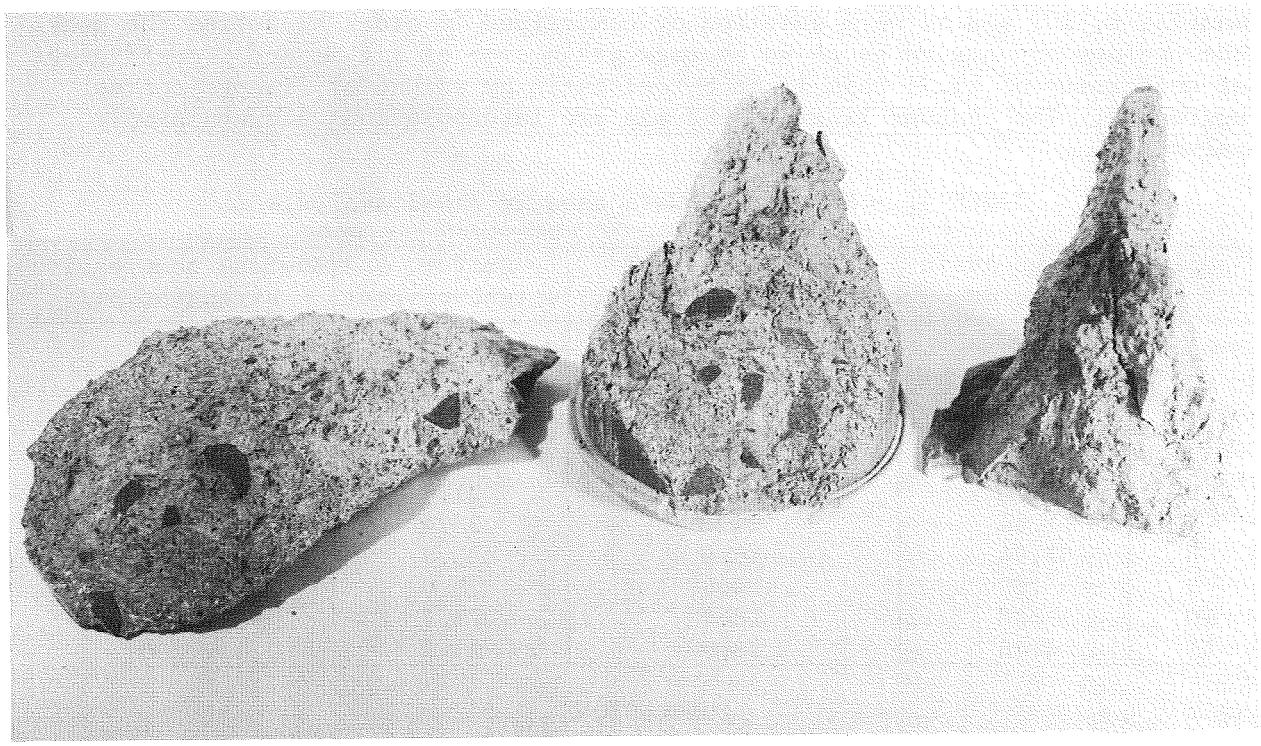

FIGURE 10. - Test Cylinder Using Well-Graded Basalt Aggregate, Failed at 10,700 psi.

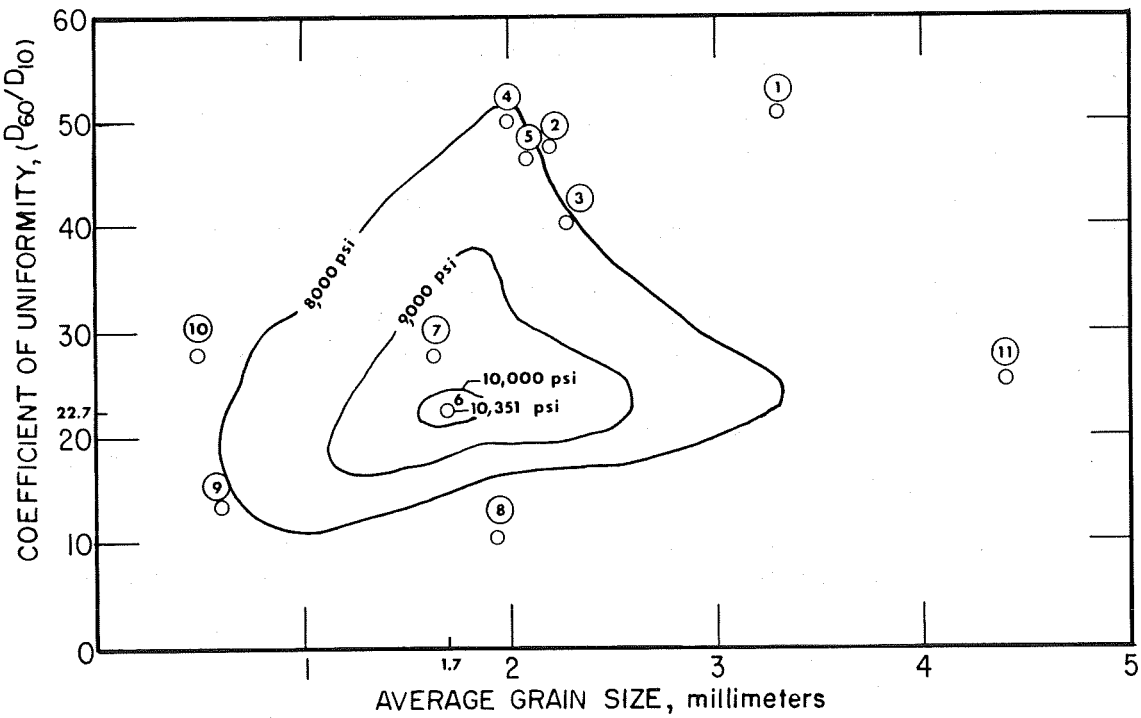

FIGURE 11. - Contour Plot of Unconfined Compressive Strength As a Function of Average Grain Size and Coefficient of Uniformity.

samples 9, 10, and 11 were adjusted or composited in order to define the peak. The optimum appears to be at an average grain size of 1.7 mm and a coefficient of uniformity of 22.7; this is an 8-to-1 ratio by weight of fine-to-coarse aggregate. The contour lines on the map are interpolations between sample values.

TABLE 4. - *Summary of data used in final analysis*

Sample	Average grain size, mm	Coefficient of uniformity (D_{60}/D_{10})	Sulfur, weight-percent	Average compressive strength, psi
1	3.30	51.1	22.6	6,400
2	2.20	47.5	22.1	7,200
3	2.30	40.7	22.1	8,000
4	2.01	50.0	22.7	8,300
5	2.10	46.7	24.0	8,700
6	1.70	22.7	21.7	10,400
7	1.64	27.7	23.4	9,200
8	1.94	10.4	23.7	6,400
9	.60	14.4	25.0	7,900
10	.48	28.4	23.7	7,500
11	4.40	26.0	21.7	7,000

CONCLUSIONS

The following conclusions are reached regarding the strength and workability of sulfur-basalt aggregate concrete:

1. To obtain maximum strength and facilitate mixing, it is essential to have a double-graded aggregate. The coarse particles furnish strength and aid in the workability of the mix. The fine particles fill the void spaces between the coarse particles, allowing surface bonding with the sulfur where it has its greatest strength.

2. Use of 10 percent minus 200-mesh material contributes toward the strength of the mixture by holding the proper amount of sulfur in suspension.

3. The problem of shrinkage is a major one, which can be overcome by puddling the top surface of the mix to prevent it from sealing over. As the sulfur contracts in the center, addition of the same mix substantially reduces the voids.

4. A gradation with excessive fines requires more sulfur and is less workable than an optimum mix of 8-to-1 ratio by weight of fine-to-coarse aggregates with 22.7 weight-percent sulfur.

5. With proper control of aggregate grain-size distribution, sulfur-basalt concretes having compressive strengths of over 10,000 psi can be produced.

6. To minimize the sublimation loss of sulfur in a lunar environment, the best approach is to insure a temperature of 20° C or less where the sulfur is exposed to high vacuum conditions. Another approach, of course, would be to seal the exposed surface with a thin coating of impervious insulating material.

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