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MECHANICAL PROPERTIES AND FREEZE-THAW RESISTANCE OF SULPHUR CONCRETE

by

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V. M. Malhotra*

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SUMMARY OF RESULTS

Sulphur concrete is composed of mineral aggregates and elemental sulphur. The optimum percentage of sulphur in the mixes studied was between 23 and 25 per cent of the weight of aggregates.

For a sulphur content at 25 per cent, 4 x 8-in. test cylinders had one-day compressive strengths between 5275 and 7600 psi. The corresponding flexural strengths were between 705 and 985 psi. There are indications of retrogression in both compressive and flexural strengths as the sulphur concrete ages.

After exposure to less than 75 cycles of freezing and thawing, the sulphur concrete prisms had shown marked deterioration. The residual flexural strength of the test prisms was between 5.9 and 14.7 per cent and the relative dynamic modulus of elasticity was generally less than 30 per cent.

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INTRODUCTION

In 1970, Canada's production of sulphur was about 5,000,000 tons, of which about 80 per cent was a by-product of cleaning Western Canada's natural (sour) gas and 20 per cent was derived from smelter operations[1]. The year-end inventory in 1970 was 3.5 million tons and it is anticipated that the inventory of elemental sulphur will accelerate to about 50 million tons by the end of 1980. As inventories increased, its price per ton fell from about $35.00 in 1968 to about $6.00 in 1972. At the lower price, uses may be found for this product. Attempts have been made to use sulphur as a construction material. Investigations by Dale[2], Dale and Ludwig[3], and Crow and Bates[4] indicate that, under controlled temperatures, sulphur can be combined with aggregates to form concrete. And, of course, materials engineers have been using sulphur capping in the testing of portland cement concrete cylinders for a long time. This investigation was undertaken to develop satisfactory mixing procedures for making sulphur concrete and to determine its mechanical properties and its resistance to freezing and thawing.

PROPERTIES OF SULPHUR

Sulphur, an element with atomic number 16 and atomic weight of 32.06, exists as rhombic and monoclinic crystals, which change reversibly at 203.7°F with the absorption of 5.386 Btu per lb. Between this temperature and its melting point, monoclinic sulphur is the stable form.

Ordinary commercial sulphur weighs between 24 and 90 lb/cu ft in bulk and melts at 234°F. Molten sulphur is straw-yellow and transparent. At its melting point, its viscosity is 12.5 centipoise*; between 248° and 320° F, its viscosity decreases linearly to 6.6 centipoise. Above 320°F, sulphur becomes dark brown and, apparently, its structure changes abruptly. For this reason, its temperature is usually maintained between 260° and 300°F.

At 20°C (68°F) the thermal conductivity of sulphur in CGS units is 0.00065(5); the corresponding value for concrete made from the usual aggregates is about 0.0030.

MATERIALS USED

Commercial sulphur, 99.9 per cent pure, was used in this investigation. The percentages retained on 200- and 325-mesh screens were 24.30 and 26.90 per cent respectively, with 48.8 per cent passing minus 325 mesh.

River gravel crushed to minus 3/4 in. was the coarse aggregate, and local sand was the fine aggregate. To keep the size distribution uniform, the sand was separated into different size fractions and recombined to specified size fractions.

The size distributions and physical properties of both the coarse and fine aggregates are given in Tables 1 and 2.

Silica flour was used as a workability aid in all the mixes.

It is to be noted that no portland cement is used in sulphur concrete.

MIX PROPORTIONS

A number of trial mixes were made to determine the best proportions of sulphur to be used. Initially, a sulphur content of 15 per cent (by weight)

* The viscosity of water is 1.0 centipoise at 68°F.

of the total aggregate was unsatisfactory because the mix was harsh and unworkable. As the sulphur was increased, the mixes became more workable and easier to handle. The mix proportions finally selected for this investigation were expressed in percentage of total weight of aggregates, as follows:

Fine aggregate	=	40 per cent
Coarse aggregate	=	60 per cent
Sulphur	=	25 per cent
Silica flour	=	6 per cent

The batch weights, using the above proportions, were as follows:

Fine aggregate	=	32.0 lb
Coarse aggregate	=	48.0 lb
Sulphur	=	20.0 lb
Silica flour	=	4.8 lb

The silica flour was used as a workability aid, and was selected because a mixture of sulphur and silica flour is commonly used for capping portland cement concrete test cylinders and because it was readily available.

MIXING PROCEDURE AND HEATING EQUIPMENT

A 2.5-cu-ft,tilting-drum, electrically operated mixer was purchased for this investigation. Its diameter was 18 inches at the top, 24 in. at the bottom over a length of 24 inches.

During initial mixes, a specially designed ring burner was used to heat the materials in the mixer (Figure 1). The ring burner had a heating capacity of over 100,000 Btu and was made of 2-in. pipe. The burner had an inside diameter of about 26.5 in. A mixture of air and propane gas was used for heating. The ring of the burner was composed of two halves for easy assembly and was provided with two mounting brackets on each half. The burner was mounted on a portable steel stand so that the tilting mixer could be moved into and out of the ring burner with ease (Figure 2).

After fine and coarse aggregates were placed in the mixer, the mixing and heating was begun. In about 10 minutes the temperature of the aggregates, as measured by a thermometer, reached about 200°F. The measured amount of sulphur was then slowly introduced into the mixer by means of a large scoop. Heating was continued until sulphur and aggregates formed a flowable mixture. This normally took about five minutes. The design of the ring burner was such that in spite of the fine controls available it was not possible to adequately regulate the inflow of the air-gas mixture and the resulting heat within close tolerances. On a few occasions, this resulted in the burning of the sulphur, very viscous mixtures, and unpleasant SO_2 fumes. Because the mixing was to be done indoors, the heating equipment was not considered satisfactory; instead the following procedure was adopted.

The aggregates for each mix were placed in tin pails and heated overnight in standard laboratory heating cabinets to about 350°F. The following morning, the coarse aggregates were placed in the tilting mixer which was then started. Immediately afterwards, about ten lb of sulphur was added so as to finely coat the aggregates. This was followed by the addition, in order of the sand, the remaining sulphur, and the silica flour. Mixing was continued for one more minuted, by which time the sulphur and aggregates had combined to form a flowable mixture (Figure 3).

Later in the program, the outside of the drum of the mixer was wrapped with asbestos sheeting to minimize the heat loss during mixing.

PREPARATION AND TESTING OF SPECIMENS

Two series of sulphur concrete mixes were made in this investigation. In the first series, three 4 x 8-in. cylinders and two 3.5 x 4 x 16-in. prisms were cast from each of 6 batches of concrete. In the second series, three 4 x 8-in. cylinders and three 3.5 x 4 x 16-in. prisms were cast from each of 5 batches of concrete.

The moulds for all specimens were filled in one continuous layer and simultaneously compacted by hand rodding using the hemisperical tip of a 24 x 0.75-inch steel rod. Extra concrete was placed on top to allow for shrinkage of the sulphur. The finish of the top surfaces of the prisms was not of great importance because they were to be tested at right angles to the direction of casting. Nevertheless, an attempt was made to obtain an even and smooth surface (Figure 4). After casting, all the moulded specimens were allowed to cool in the laboratory air for a couple of hours and were then demoulded. At the end of selected curing periods at room temperature, the specimens were tested. Before testing, the top quarter-inch of each test cylinder was sawn off to remove the excess material and to obtain a plane surface. The cylinders were tested in compression* on an Amsler testing machine (capacity 600,000 lb) and the prisms were tested in flexure** on a Tinius Olsen testing machine (capacity 60,000 lb) in accordance with ASTM Standard methods.

DURABILITY STUDIES

In order to determine their resistance to frost action, the Series II prisms were exposed to accelerated cycles of freezing and thawing.

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* ASTM Standard Method C 39-72
** ASTM Standard Method C 78-69 (1972)

Freezing and Thawing Procedure

After 57 to 60 days of storage at room temperature, two prisms specimens from each batch of Series II were exposed to repeated cycles of freezing in air and thawing in water according to ASTM Standard Test C 666-71.

The automatic freeze-thaw unit* used performs 8 cycles per day. One complete cycle from 40 ± 3°F to 0 ± 3°F and back to 40 ± 3°F requires about three hours. Weight, resonance frequency, and pulse velocity measurements were made on all prism specimens before the freeze-thaw testing. This was followed by placing two prisms from each batch in the freeze-thaw unit, and retaining the third prism as the "reference prism".

During freeze-thaw cycling, a visual check was kept on the specimens. When the specimens had shown sufficient deterioration, the test was discontinued and the weight, resonance frequency, and pulse velocity were again determined. These tests were also performed on the "reference" prisms. Following this, all the prisms were tested in flexure by the method previously described.

The relative dynamic modulus of elasticity for the purpose of discussion was calculated from the following equation, given in ASTM Standard C 666-71.

$$P_c = \frac{N_1^2}{N^2} \times 100 \text{ per cent}$$

where N = fundamental longitudinal frequency at zero cycles of freezing and thawing .

N_1 = fundamental longitudinal frequency after the freeze-thaw test.

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*Manufactured by the Canadian Ice Machine Company Ltd., Toronto, Ontario.

TEST RESULTS AND THEIR ANALYSIS

Eleven batches of sulphur concrete were made, and 30 prisms and 28 test cylinders were tested in this program. The strength test results together with the results of freeze-thaw studies are summarized in Tables 3 to 6. Where possible, standard deviation and coefficient of variation for the test data were calculated and these are shown in Tables 7 and 8.

The densities of 4 x 8-in. test cylinders, just before testing, are shown in Table 4.

DISCUSSION OF TEST RESULTS

Best Percentage of Sulphur as a Binder

The results indicate that the best percentage of sulphur in the mix is between 23 and 25 per cent, by weight of aggregates. Of course, this percentage is only true for the type, size, and grading of aggregates used. The trial mixes, containing 15 per cent by weight of sulphur, were very harsh and the test specimens had poor finish. There was clear visual evidence that the specimens were deficient in binder material. As the percentage of sulphur was increased, the test specimens made from the resulting mixes were more homogeneous and had good finishing characteristics (Figure 5). In trial mixes containing 30 per cent of sulphur, there was some segregation because of an excessive amount of binder.

Workability of Sulphur-Aggregate Concrete Mixes

The workability of sulphur-aggregate concrete was generally poor, even at the best sulphur content. The addition of silica flour considerably increased the workability and handling properties. Unfortunately, apart from the visual examination, there was no means of comparing the workabilities. The various workability tests which are available for portland cement concrete are not applicable to sulphur concrete. Based on this limited laboratory experience, it is believed that the use of admixtures will help in the handling of sulphur concrete. Other additives which may be promising in this regard are fly ash and diatomiceous earth.

Casting of Test Cylinders

As mentioned earlier, sulphur shrinks on cooling and this leaves large cavities and an uneven surface on top of the cylinders (Figure 6). To overcome this problem, extra sulphur concrete was heaped on top of the test cylinders during casting; after cooling, or just before testing, this was sawn off to obtain a smooth surface. A better solution appears to be to attach a collar on top of each cylinder similar to the collars on the compaction moulds used for testing soils. After the sulphur concrete has hardened, or just before testing, the extra concrete can be neatly sawn off.

Strength of Sulphur Concrete

The compressive and flexural strengths of the test specimens of sulphur concrete are excellent. The compressive strength at one day*, using the best percentage of sulphur, exceeds 5000 psi and this is comparable to 28-day strengths of portland cement concrete that contains about 550 lb cement per cu yd. In the latter case, the specimens are cured under standard temperature and humidity, whereas in the former case, the specimens are cured at room temperature.

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* It has been reported (9) that at 6 hours sulphur concrete attains 88 per cent of its ultimate strength, which occurs somewhere between 1 and 28 days.

For the sulphur concrete specimens tested, the modulus of rupture was between 10.7 and 14.6 per cent of the compressive strength. There is no evidence of decrease in these percentages with an increase in compressive strength. Comparatively, for portland cement concrete, the above percentage is between 11 per cent at 9000 psi and 23 per cent at 1,000 psi compressive strength level.

The specimens tested at age one day had compressive strengths between 5275 and 7600 psi but those tested between 72 and 77 days had strengths between 4725 and 6180 psi, though the mix proportions were the same in each case. Unfortunately, it was not possible to make a large batch and to cast a number of test specimens to be tested at various ages. This would have eliminated the batch effect which may be contributing to the difference in strength at 1 and 72 to 77 days. However, additional sulphur concrete mixes are being tested to investigate this aspect.

Within-Batch and Between-Batch Variation

The within-batch coefficients of variation (C.V.) for compressive strength results at one day (Table 7) are between 1.8 and 9.7 per cent with an average value of 4.8 per cent; the corresponding value for flexural strengths are between 1.1 and 15.6 with an average value of 6.4 per cent. The above values are comparable to within-batch variation of test results for portland cement concrete. However, the within-batch C.V.'s for compressive strength for Series II mixes (Table 8) are between 4.3 and 26.1 per cent with an average value of 12.0 per cent. This spread is so wide because sulphur concrete is difficult to cast into identical specimens.

The compressive strengths for test specimens cast from Series I mixes are between 5275 and 7600 psi, giving a between-batch C.V. of 15.8 per cent. The corresponding values for Series II mixes are between 4820 and 6180 psi, with a C.V. value of 12.8 per cent. These high values once again underline the difficulty of reproducing the same batch of sulphur concrete repeatedly.

Elastic Properties of Sulphur Concrete

During handling and testing, the test specimens made with sulphur concrete appeared to be more brittle than similar specimens of portland cement concrete. In compression testing, the breakdown of the structure of the concrete was quite audible. Young's modulus of elasticity and creep of the sulphur concrete were not determined. However, it has been reported[(6)] that the modulus of elasticity of sulphur concrete made with limestone aggregate was of the order of one million psi and creep was 0.04 per cent at 14 days.

Test specimens immediately after strength tests are shown in Figure 7.

Exposure of Sulphur Concrete Prisms to Freeze-thaw

The test results (Table 5) show that the prisms had been extensively damaged after exposure to less than 75 cycles of freezing and thawing (Figure 8). In some cases, due to the extremely deteriorated conditions of the prisms, no ultrasonic pulse velocity and longitudinal resonant frequency readings were possible. In general, after the freeze-thaw test, the ultrasonic pulse velocity and longitudinal resonant frequency readings were less than 1/3 and 1/2 respectively of the readings at the commencement of the test.

The residual flexural strength of the test prisms exposed to freezing and thawing were between 5.9 and 14.7 per cent (Table 6) which, in effect, means that the prisms had lost all their flexural strength.

The freeze-thaw test used for the prisms under investigation is the same as that used for portland cement concrete. As the thermal conductivity of sulphur concrete is lower than that of portland cement concrete, it is possible that the centre of sulphur concrete prisms may not have reached either temperature limit. In spite of this, the prisms did show extensive damage.

Whether the prisms would have suffered less or more damage in a slower test of only two cycles per day (instead of 8 as in the present test) is a matter of conjecture. Further investigations are indicated in this direction.

The relative dynamic modulus of elasticity was between 19.4 and 40.4 per cent but was generally less than 30 per cent, indicating once again the poor conditions of prisms after the test.

Attempts were made to improve the freeze-thaw resistance of sulphur concrete by polymer impregnation. Four two-in. cubes of sulphur concrete were made using the same mix proportions as used in Series I and II. One side of the cubes was lapped to expose the aggregates and to remove the coating of sulphur. The cubes were then sent to the Department of Civil Engineering, Queen's University, Kingston, where two of them were impregnated with methyl methacrylate as the monomer. Following polymerization, the two test cubes together with the two control cubes were subjected to freeze-thaw cycling. The results were not encouraging; the impregnated test cubes had started to deteriorate at about 80 cycles of freezing and thawing; by this time of course, the control cubes had wide-open cracks (Figure 9). At the end of 100 cycles of freezing and thawing, both the test cubes and the control cubes had badly deteriorated.

The poor performance of the impregnated specimens is due to the fact that the weight of the polymer impregnated was only 0.37% by weight of the sulphur concrete cube. This low impregnation was probably due to the sulphur specimens having a rather low volume fraction of pores that can be penetrated by methyl methacrylate, i.e. the pore structure is insufficiently continuous to permit much impregnation. Thus sulphur concrete cannot be polymerized by the conventional techniques used for polymer concrete[7].

Duecker[8] has reported some improvement in the properties of sulphur, including resistance to freezing and thawing, by the use of chemical admixtures.

GENERAL COMMENTS

From the work reported in this investigation and that reported by others[2,3,4,6,9], it is seen that high-strength material can be produced by combining mineral aggregates and sulphur under controlled temperature conditions. Sulphur concrete appears to have satisfactory mechanical properties, but very little is known about its modulus of elasticity, creep shrinkage, and its behaviour under wetting and drying and under repeated loading conditions. This investigation has shown its limitations with respect to freeze-thaw resistance. Before sulphur concrete can be considered suitable for use as a structural material, much more must be learned about its creep behaviour under sustained loading and about its other elastic parameters.

It is believed that creep of sulphur concrete will be excessive at later ages. This, combined with its low modulus of elasticity, brittleness, and probable retrogression in strength at later ages, would be a serious obstacle in its use as a structural material. The mechanical, elastic and fire resistant properties of sulphur concrete may be improved by chemical and fibrous additives[3,9]. However, even if sulphur concrete can be used in specialized applications as a structural material, and it has been tried[10], the amount of sulphur used in such instances will be so small as to hardly cause a dent in the sulphur inventory. What perhaps is needed are applications where sulphur concrete can be used in massive quantities. The use of sulphur concrete as a sub-base material for highway construction may be one such application.

CONCLUDING REMARKS

Elemental sulphur can be combined with mineral aggregates to produce high-strength concrete. The best content of sulphur in the mixes studied seems to be between 23 and 25 per cent by weight of aggregates.

The most satisfactory way to make sulphur concrete in a laboratory is to add sulphur to ovenheated aggregates. External application of heat to the drum of the concrete mixer is neither desirable nor practical.

The test specimens of sulphur concrete had high compressive and flexural strengths. However, there were indications of retrogression in strength with age.

The high within-batch and between-batch variations in compressive strength stem from the mixing and casting difficulties.

Sulphur concrete has poor resistance to repeated cycles of freezing and thawing. This would discourage its use as a structural concrete. However, this should not preclude its applications where sulphur concrete is not exposed to freeze-thaw conditions.

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TABLE 1

Grading of Aggregates

Coarse Aggregate			Fine Aggregate	
Sieve size	Cumulative percentage retained		Sieve size	Cumulative percentage retained
	Grading A*	Grading B**		
3/4 in.		-		
1/2 in.	0	50.0		
3/8 in.	50.0	85.0		
No. 4	100.0	100.0	No. 4	0
			No. 8	10.0
			No. 16	32.5
			No. 30	57.5
			No. 50	80.0
			No. 100	94.0
			Pan	100.0

* Used for Mix No. 1
** Used for Mix No. 2 to 11 inclusive

TABLE 2

Physical Properties of Coarse and Fine Aggregates

	Crushed gravel	Natural sand
Specific gravity	2.72	2.70
Absorption	0.40	0.50

TABLE 3

Summary of One Day Strength Test Results - Series I

Mix No.	Date Cast	Density of 4 x 8-in. cylinders lb/cu ft	Strength Test Results	
			Compressive strength of 4x8-in. test cylinders, psi	Flexural strength of 3.5x4x16-in. prisms, psi
1	May 24, 1972	154.3	6370 7685 7725 Av = 7260	940 860 Av = 900
2	May 24, 1972	155.5	5175* 5375 Av = 5275	780 625 Av = 705
3	May 25, 1972	153.5	7270 7390 8140 Av = 7600	965 1005 Av = 985
4	May 26, 1972	155.9	5615 5695 5495 Av = 5600	815 one prism highly honeycombed and discarded
5	May 26, 1972	151.8	5575 5795 6170 Av = 5845	909 895 Av = 905
6	May 29, 1972	153.7	7530 7165 Av = 7350	**

*Third specimen highly honeycombed.
**Prisms used for freeze-thaw test, see Table 5.

TABLE 4

Summary of Strength Test Results - Series II

Mix No.	Date Cast	Date Tested, 1972	4 x 8-in. Cylinders	
			Density, lb/cu ft	Compressive Strength, psi
7	May 30	August 15	154.9	5040 7325 Av = 6180
8	May 31	August 15	156.9	5750*
9	June 1	August 15	157.0	5550 5255 5730 Av = 5510
10	June 1	August 15	154.0	4280 4325 5575 Av = 4725
11	June 5	August 15	155.7	5510 4125 Av = 4820

* Only one cylinder tested.

TABLE 5

Test Results on Prisms Subjected to Freezing and Thawing

Mix No.	Number of Freeze-thaw Cycles	Test Results				Description of Test Prisms at the End of Freeze-Thaw Cycling
		Weight, lb	Longitudinal Resonant Frequency cyc/sec	Ultrasonic pulse velocity, ft/sec	Flexural Strength, psi	
6	0	21.619 21.619	4910 4990	15,080 15,070		
	75	21.952 22.030	* *	* *	* *	Both prisms had been damaged very severely and were at the point of disintegration.
7	0	21.281 20.580	5210 5040	15,240 15,280		
	65	20.092 20.631	* 3200	* *	45 75	One end of the prism was broken. This prism showed severe deterioration
8	0	21.795 20.772	5200 5220	15,555 15,555		
	65	21.855 20.825	2680 2400	4,440 4,660	30 85	Both prisms showed signs of disintegration.
9	0	20.780 21.030	5230 5240	15,555 15,460		
	65	20.848 21.120	2300 2370	4,490 3,920	55 60	Both prisms showed signs of disintegration.
10	0	20.452 20.270	5050 5100	15,110 15,110		
	65	20.490 20.332	2750 3100	5,270 6,050	35 45	Both prisms showed signs of serious deterioration.
11	0	20.810 21.342	5200 5200	15,280 15,460		
	65	20.900 21.400	2450 3000	3,510 4,370	35 45	Both prisms showed signs of serious deterioration.

* No readings possible.

TABLE 6

Summary of Flexural Strength Test Results

Mix No.	Reference Prisms		Freeze-Thaw Prisms		
	Age, days	Strength,* psi	Number of Freeze-thaw cycles	Average Strength,** psi	Residual Strength, per cent
7	77	702	75	60	8.5
8	76	407	65	60	14.7
9	75	445	65	55	12.1
10	75	675	65	40	5.9
11	74	495	65	40	8.1

* Only one prism was tested.

** Average of two test results.

TABLE 7

Within-Batch Standard Deviation and Coefficient of Variation - Series I

Mix No.	Compressive Strength			Flexural Strength		
	Average Strength, psi	Standard Deviation, psi	Coefficient of Variation, per cent	Average Strength, psi	Standard Deviation, psi	Coefficient of Variation, per cent
1	7260	771	9.7	900	56	6.3
2	5275*	141	2.7	705	110	15.6
3	7600	471	6.2	985	28	2.9
4	5600	101	1.8	815**	-	-
5	5845	301	5.1	903	10	1.1
6	7350*	258	3.5	-	-	-

* Average of two test results only.
** Only one prism tested.

TABLE 8

Within-Batch Standard Deviation and Coefficient of Variation - Series II

Mix No.	Average Compressive Strength, psi	Standard Deviation, psi	Coefficient of Variation, per cent
7	6180*	1615	26.1
8	5780**	-	-
9	5510	240	4.3
10	4725	735	15.5
11	4820*	980	20.3

*Average of two test results only.
**One cylinder tested only.

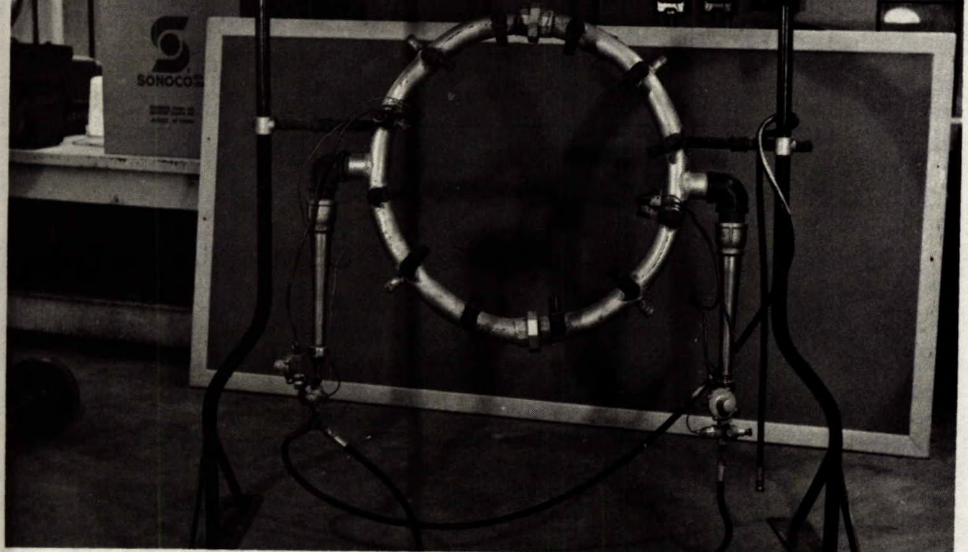

Figure 1. A view of the ring burner.

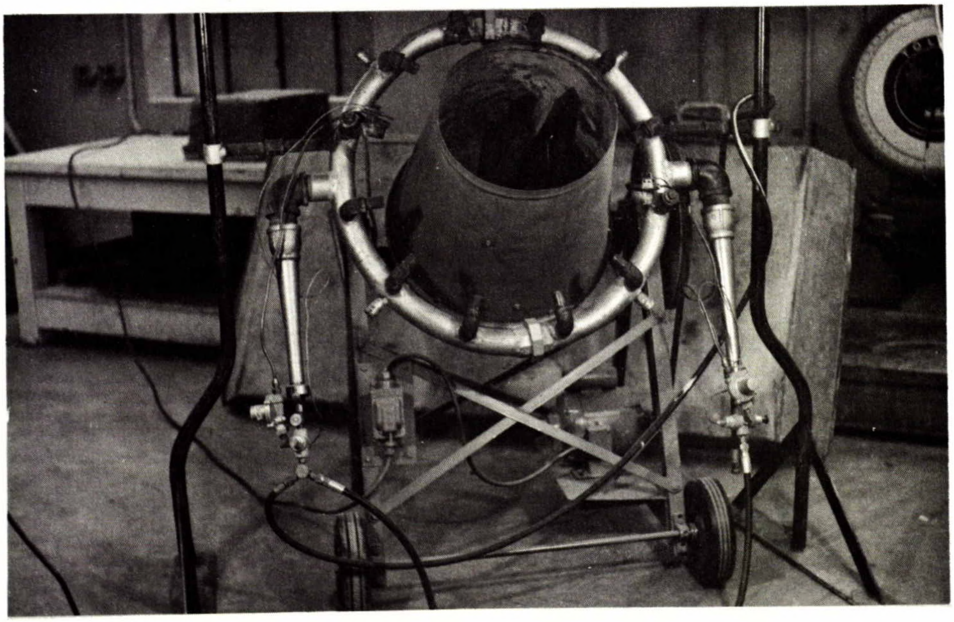

Figure 2. A view of the ring burner with mixer in position.

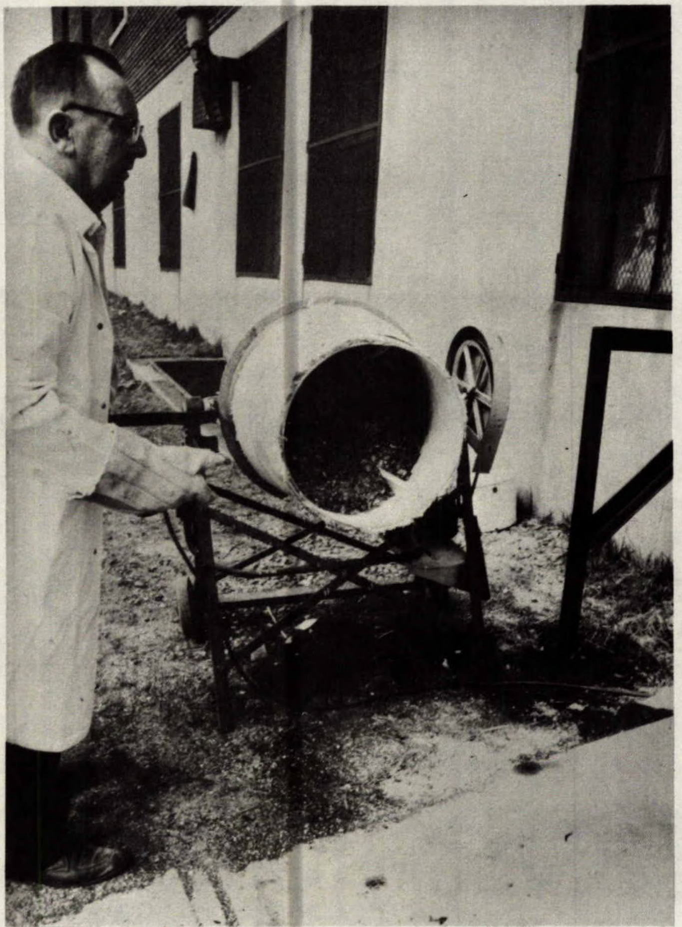

Figure 3. Sulphur concrete immediately after mixing.

During casting

After demoulding

Figure 4. A view of test specimens during casting and after demoulding.

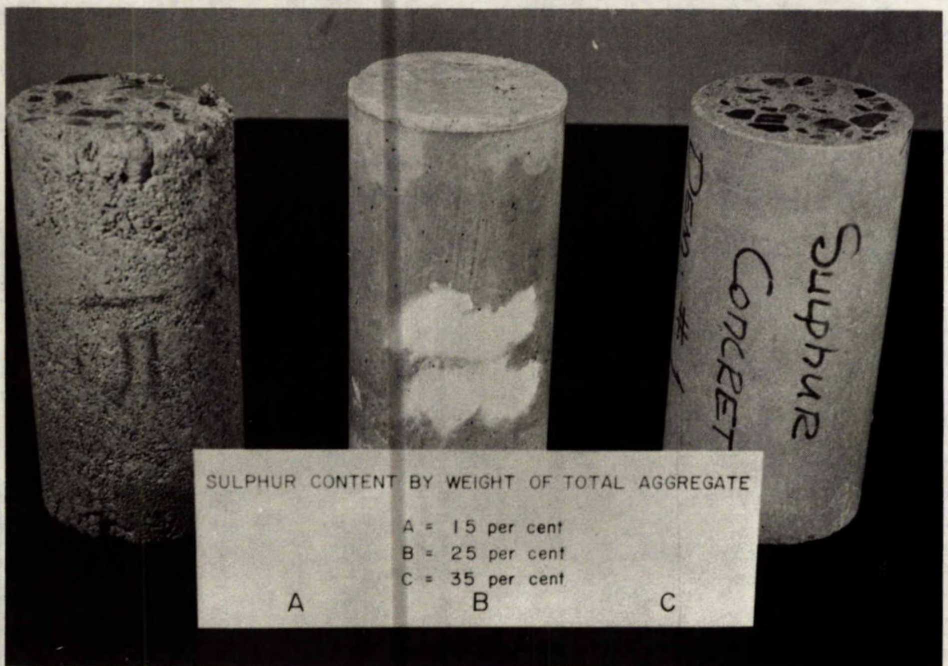

Figure 5. Test cylinders with varying percentages of sulphur.

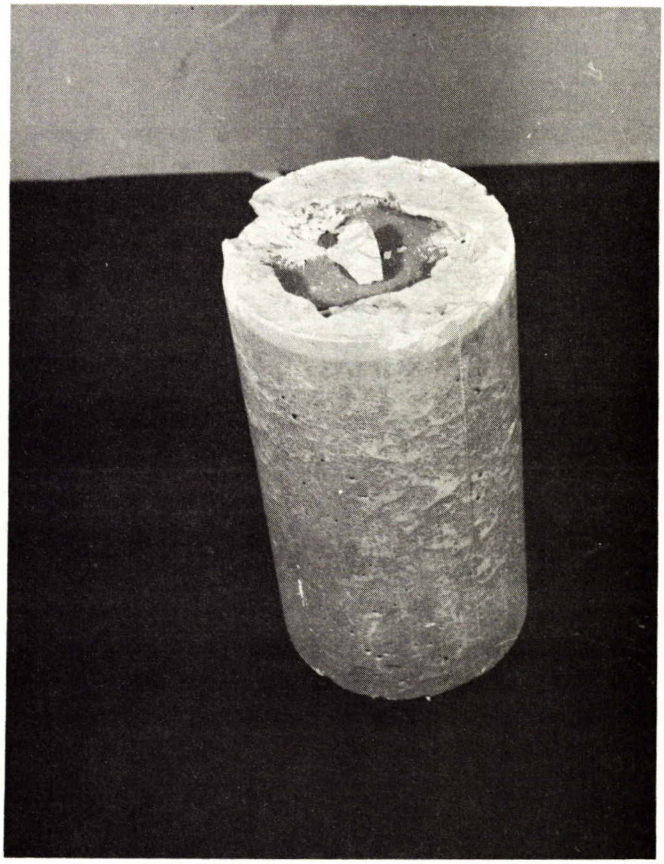

Figure 6. Test cylinder showing cavities at top due to shrinkage.

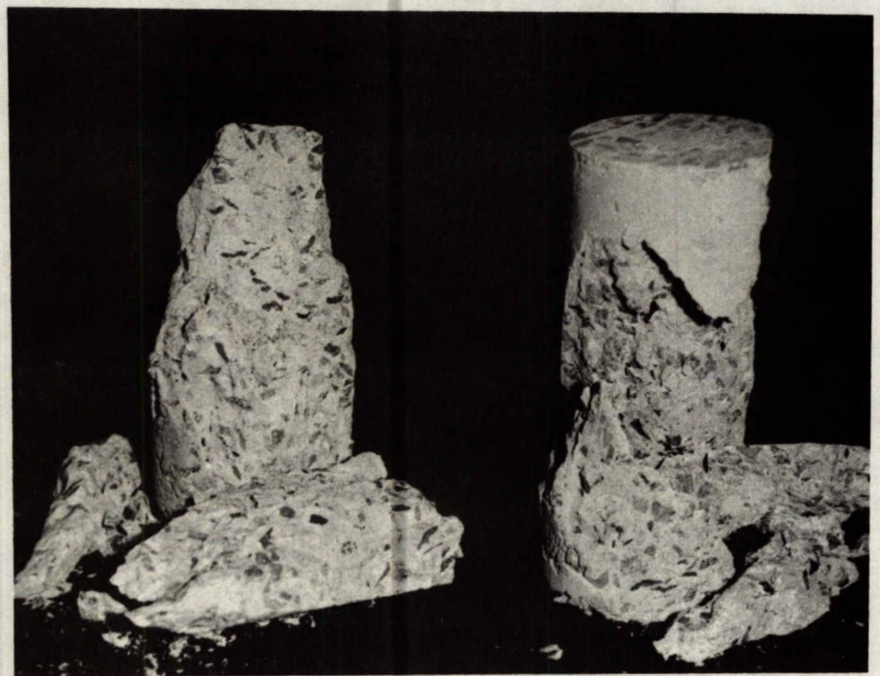

Figure 7. A view of test specimens immediately after strength tests.

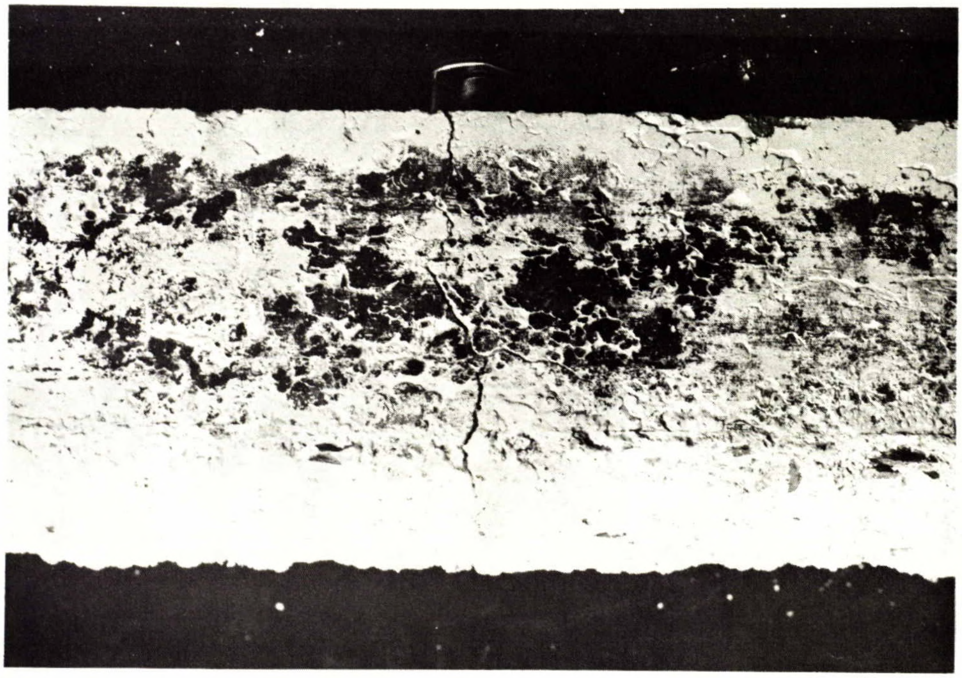

Figure 8. Sulphur concrete prism after exposure to freeze-thaw cycling.

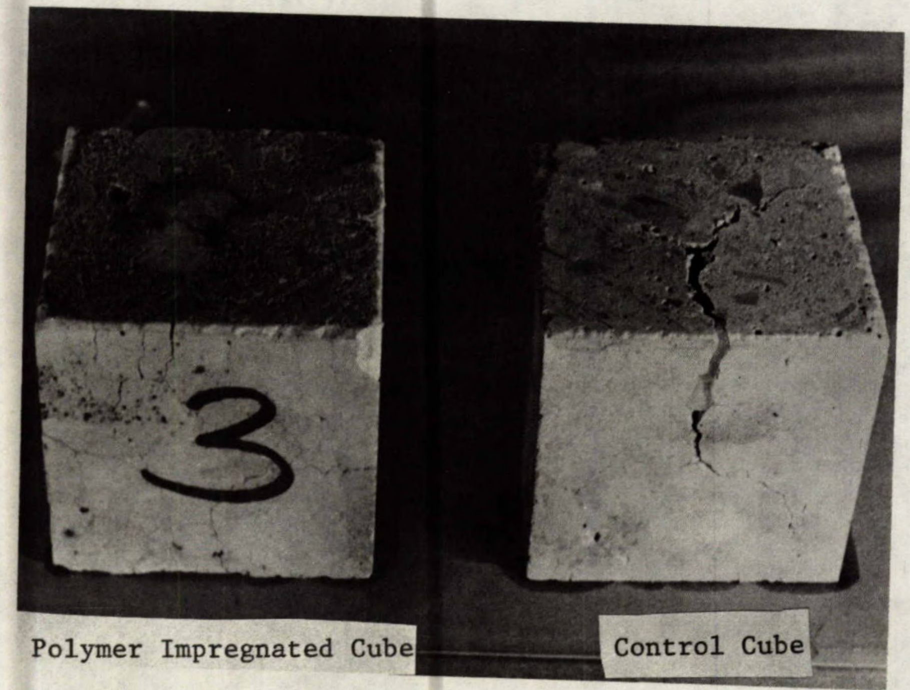

Figure 9. Polymer impregnated and control test cubes after freeze-thaw exposure.