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Guide for Mixing and Placing Sulfur Concrete in Construction

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Sulfur concrete construction materials are now used in many specialized applications throughout industry and transportation. They are currently used primarily in areas where conventional materials like portland cement concrete (PCC) fail such as acidic and saline chemical environments. These new construction materials are thermoplastic and achieve compressive strength in excess of 9000 psi within one day of casting. The materials are impervious to moisture permeation and extremely resistant to attack by mineral acids and salts. This guide includes information on historical background as well as a user's guide to sulfur concrete construction.

Keywords: acid resistance; binders (material); **concrete construction;** corrosion resistance; cyclopentadiene compounds: history; permeability; polymerization; sulfur concrete; thermoplasticity.

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CHAPTER l-HISTORICAL BACKGROUND

l.l-Introduction

Sulfur concrete is a relatively new material, and although similar in final appearance to portland cement concrete, its manufacture, handling, use, and testing are different. The purpose of this guide is to familiarize engineers, contractors, manufacturers, and users with sulfur concrete and its applications and to assist them in selecting proper materials, mixture proportions, and properties for optimum use of sulfur concrete.

Sulfur concrete is a thermoplastic material prepared by hot-mixing sulfur cements and mineral aggregates. Sulfur concrete solidifies and gains strength rapidly upon cooling. As with other concrete materials, such as portland cement concrete and asphaltic concrete, sulfur concrete is a generic term for a range of products that vary with aggregates, sulfur cements, and proportions used. By using sulfur cement binders and aggregates that are not attacked by many mineral acid and salt solutions, high-strength corrosion-resistant sulfur concrete can be produced for use in certain applications where other construction materials deteriorate rapidly. Sulfur concretes are generally not resistant to alkalis or oxidizers but exhibit excellent performance in many acidic and salt environments.

Early sulfur concrete products, prepared with unmodified sulfur as the binder, were plagued with durability problems. While materials with excellent mechanical strength properties were produced, in actual use they deteriorated and failed after a relatively short period of time. The development of modified sulfur cements, however, increased the durability of sulfur concrete and made its use as a construction material much more feasible.

When modified sulfur cement is used as the binding agent with appropriate aggregates, the resulting sulfur concrete has shown some unique properties:

a) high strength and fatigue resistance;

b) excellent corrosion resistance against most acids and salts; and

c) extremely rapid set and strength gain.

One of sulfur concrete's greatest uses is for industrial floors that are exposed to highly corrosive substances.

1.2-Early research on sulfur

The utilization of sulfur as a molten bonding agent dates back to prehistoric times.[1] During the 17th century, sulfur was used to anchor metal in stone and similar practices are still being used in Latin America.[2]

The post World War I (1919) demand for sulfur led to the opening of the "Big Dome" sulfur deposit near Matagorda, Texas. This operation doubled the production of sulfur in the United States and resulted in a surplus.

Bacon and Davis in 1921 reported on the projected uses of sulfur in construction materials to utilize the surplus sulfur.[3] They tested many suggested additives for modifying the properties of sulfur for specific uses and found almost all of them unsuitable. In their work, they found that a mixture of 60-percent sand and 40-percent sulfur produced an acid-resistant material with excellent strength.

Kobbe, in 1924, reported on the acid-resistan properties of materials prepared from sulfur and coke.[4]

Duecker, in 1934, found that the 60 percent sand and 40 percent sulfur product of Bacon and Davis increased in volume on thermal cycling with a loss in flexural strength.[5] Duecker was able to retard both the tendency for volume increases and the resulting loss of strength on thermal cycling by modifying the sulfur with an olefin polysulfide. The use of additives to prepare more stable cements and products led to more industrial acceptance and more research and development on means of improving sulfur products for use as acid-resistant mortars and grouts.

McKinney, in 1940, outlined testing methods for sulfur materials that had been found satisfactory at the Mellon Institute.[6] Many of these methods have been adopted and are found in ASTM specifications for chemical-resistant sulfur mortar.[7]

The developments that led to the ability to modify sulfur and produce a more durable product brought increased interest in research towards commercial activities. The research mainly has been divided into two categories: sulfur concrete and sulfur-infiltrated concrete. This report concentrates on the use of sulfur concrete because the latter has not as yet reached large-scale commercial usage.

1.3-Recent research on sulfur concrete

Major advances in the development of sulfur concrete have taken place in the last decade. Research was based on the premise that for sulfur concrete to be a viable construction material, its durability would have to be improved, improved sulfur cements would have to be developed, and better mixture designs would have to be developed for the production of uniform products on a routine basis.

In the late 1960s, Dale and Ludwig pioneered the work on sulfur-aggregate systems, pointing out the need for well-graded aggregates to obtain optimum strength.[8,9] This work was followed by the investigations of Crow and

Bates on the development of high-strength sulfur-basalt concretes.[10] The United States Department of the Interior's Bureau of Mines and The Sulphur Institute (Washington, D.C.) launched a cooperative program in 1971 to investigate and develop new uses of sulfur.[11] At about the same time, the Canada Centre for Mineral and Energy Technology (CANMET), and the National Research Council (NRC) of Canada initiated a research program in the development of sulfur concrete.[12-14] This was followed by work at the University of Calgary, Alberta.[15]

In 1973 the Sulfur Development Institute of Canada (SUDIC), jointly founded by the Canadian Federal Government, Alberta Provincial Government, and the Canadian sulfur producers, was established to develop new markets for the increasing Canadian sulfur stockpile. In 1978 CANMET and SUDIC sponsored an international conference on sulfur in construction.[16] Also during this period a number of investigators including McBee and Sullivan, Malhotra, Vroom, Yuan, Loov et al., Gregor and Hackl, Diehl, Lee, and others published a number of papers and reports dealing with various aspects of sulfur and sulfur concrete.[17-30] All of these activities led to an increased awareness of the potential use of sulfur as a construction material.

While sulfur concrete materials could be prepared by hot-mixing unmodified sulfur and aggregate, durability of the resulting product was a problem. Unmodified sulfur concretes failed when exposed to repeated cycles of freezing and thawing, humid conditions, or immersion in water. Research was aimed at establishing reasons for failure of these sulfur concrete materials and determining means of preventing failures.

When unmodified sulfur and aggregate are hot-mixed, cast, and cooled to prepare sulfur concrete products, the sulfur binder, on cooling from the liquid state, first crystallizes as monoclinic sulfur (S_β) at 238 F (114 C) with a volume decrease of 7 percent. On further cooling to below 204 F (96 C), the S_β starts to transform to orthorhombic sulfur (S_α), which is the stable form of sulfur at ambient temperatures.[31] This transformation is rapid, generally occurring in less than 24 hrs. As the S_α form is more dense than S_β, high stress is induced in the material by the solid sulfur shrinkage. Thus, the sulfur binder can become highly stressed and can fail prematurely. The expansion of mortar prisms made with sulfur sand is an example of the failure of a sulfur product through stress relief by thermal cycling; this was observed by Duecker.

It was necessary to develop an economical means of modifying the sulfur so that the sulfur concrete product would have good durability. While olefinic polysulfide additives had shown promise in these applications, their costs were deemed prohibitive for use in preparing sulfur concrete for large-scale construction uses.

In 1973, a process was developed by A.H. Vroom with assistance from the National Research Council of Canada and A. Ortega of McGill University, Montreal.[14] This process involved modifying sulfur by reacting it with olefinic hydrocarbon polymers.[29,30] It was also discovered that a similar reaction would yield a sulfur soluble polymer concentrate. The resulting sulfur concrete was first produced for commercial use in Calgary, Alberta, in 1975.[32]

Modification of sulfur by reaction with dicyclopentadiene (DCPD) has been investigated by many researchers, but its practical use in commercial applications has been limited because the reaction between sulfur and DCPD is exothermic and requires close control. Also, the DCPD modified sulfur cement is unstable when exposed to high temperatures (greater than 285 F [140 C]), such as when mixing with hot aggregates, and may react further to form an unstable sulfur product which reverts back from the S_β to the S_α form. McBee and Sullivan solved this problem through development of a process for preparing modified sulfur cement that is stable in the S_β form and is not temperature sensitive in the mixing temperature range for producing sulfur concrete.[20] This process utilizes the controlled reaction of cyclopentadiene (CPD).

Other researchers have reported methods for treating sulfur for use in sulfur concretes. They include: Leutner and Diehl[33] using DCPD; Gillott et al.[34] using crude oil and polyol additives; Schneider and Simic[35] using DCPD or a glycol; Woo[36] using phosphoric acid to improve freeze-thaw resistance; and Nimer and Campbell[37] using organosilane to improve water stability. In addition, Gregor and Hackl[38] have reported on laboratory design tests for DCPD modified sulfur concrete products; Bright et al.[39] on modified sulfur systems; and Bordoloi and Pierce[40] on stabilizing sulfur with DCPD.

Since 1976, commercial production and installation of corrosion-resistant sulfur concrete has been increasing continually.[41-47] The sulfur concrete materials are being either precast or installed directly in industrial plants where portland cement concrete materials fail from acid and salt corrosion. Typical installations are as floors, slabs on grade, overlays, curbs, walls, trench drains, sump pits, tanks, electrolytic cells, pump bases, column piers, foundations and pipes.[42]

The descriptions in this report of sulfur concrete materials are based largely on published reports and experience gained during commercial installations.

1.4-Advantages and concerns with sulfur concrete

The main advantage of sulfur concrete is its use as a highly durable replacement for construction materials, especially portland cement concrete, in locations within industrial plants or other locations where acid and salt environments result in premature deterioration and failure of portland cement concrete.

There are several advantages in using sulfur concrete for construction in areas exposed to highly corrosive acids. While ultimate life or durability of sulfur concrete has not been completely established in many end use applications, enough evidence of its corrosion resistance and durability has been accumulated to show that it has several times the life of other construction materials now being used in corrosive environments.[26] Other advantages of sulfur concrete are its fast setting time and rapid gain of high strength. Since it achieves most of its mechanical strength in less than a day, forms can be removed and the sulfur concrete placed in service without a long curing period.

Generally, sulfur concrete has the following useful characteristics:

1. Sulfur concrete's tensile, compressive, and flexural strengths" and fatigue life[48] are greater than those obtained with normal portland cement concrete. Fig. 1.4 illustrates compressive strength versus age for the two materials. Normal portland cement concrete is a material prepared with Type 1 cement (six sacks per cubic yard) and aggregates 0 to 1% in.[49]

2. Sulfur concrete shows excellent resistance to attack by most acids and salts, some at very high concentrations.[26]

3. Sulfur concrete sets very rapidly and achieves a minimum of 70 to 80 percent of ultimate compressive strength within 24 hr.[27]

4. Sulfur concrete can be placed year-round, in below-freezing temperatures.

5. Sulfur concrete exhibits very low water permeability.[24]

The handling, mixing, and use of sulfur concrete can be accomplished with proper concern for product safety.[27,28,42,47] This is a general report and it does not purport to address all of the safety considerations associated with the use of sulfur concrete.

As with any other construction material, certain measures must be taken with sulfur concrete to insure safe handling in its preparation and use. Sulfur concrete should be produced within its recommended mixing temperature range of 260 to 300 F (127 to 141 C) to minimize emissions. Adequate ventilation during construction operations and normal precautions for handling hot fluid materials (proper protective clothing, eye protection, gloves, and hard hats) should be observed. Practices for safe handling of both solid and liquid sulfur have been established by the National Safety Council50,51 and should be observed when preparing and handling sulfur concrete.

If exposed to temperatures above the melting point of the sulfur cement, sulfur concrete will loose strength. Also, sulfur concrete must be used in applications consistent with its strength and thermoplastic properties.

CHAPTER 2-USER'S GUIDE TO SULF'UR CONCRETE CONSTRUCTION

2.1-Modified sulfurs and aggregates

2.1.1 ***Modified sulfur cements-The*** need for using modified sulfur cements in sulfur concrete manufacture has been recognized since the 1930s. Sulfur cements are modified to improve the stability and durability and reduce the expansion-contraction of sulfur concrete during thermal cycling.

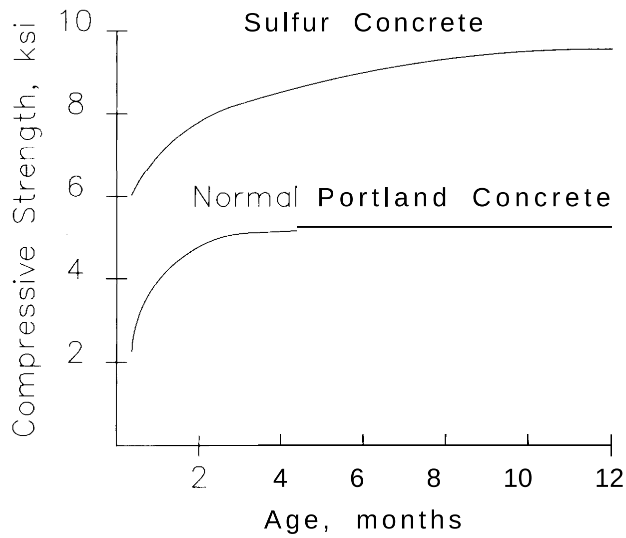

Fig. 1.4-Compressive strength versus age for sulfur concrete and normal portland cement concrete

There are several methods that can be used to produce modified sulfur cements. Two methods are currently used in North America to produce sulfur cements that are marketed under different trade names.

Method 1 is based on the polymeric reaction product of sulfur with a modifier containing equal parts of cyclopentadiene oligomer and dicyclopentadiene.[22] The composition and properties of Method 1 modified sulfur cement are as follows:[52]

Sulfur, percent by weight	95.0 ± 1.0
Carbon, percent by weight	5.0 ± .5
Hydrogen, percent by weight	.5 ± .05
Specific gravity at 77 F (25 C)	1.90 ± .02
Viscosity, centipoises at 275 F (135 C)	25 to 100

Method 2 utilizes a modified sulfur concentrate prepared by combining sulfur with olefinic hydrocarbon polymers such as Escopol.[30] This concentrate is then mixed with locally available pure sulfur in the ratio of 10 parts of sulfur to 1 part of concentrate, by weight. The concrete used with Method 2 modified sulfur has the following approximate composition:

Sulfur, percent by weight	80
Carbon, percent by weight	18
Hydrogen, percent by weight	2

Both methods of modifying sulfur give extremely long shelf life in the solid state. If stored in the molten state, both modifiers may continue to react and produce inferior concretes. The users should follow the recommendations of the material supplier for the time limits that the modified sulfur can be held in the liquid state.

Testing methods for modified sulfur cement are:

1. Sulfur and carbon are determined by combustion of

the sulfur cement using a carbon/sulfur analyzer.[28]

2. Specific gravity measurements are made in accordance with ASTM D 70. Measurements are made at 77 F (25 C).

3. Viscosity is determined at 275 F (135 C) using a rotating spindle-type viscometer equipped with an electrically heated cell and temperature controller.[28]

2.1.2 *Aggregates*-Selectionof quality aggregates, appropriate for each application, is essential for sulfur concrete materials. Aggregates should meet ASTM C 33 specifications with respect to durability, cleanliness, and limits of deleterious substances. The aggregates should be resistant to chemical attack by the environment in which they are to be used. For example, quartz aggregates are suitable for use in both acidic and salt environments, whereas limestone aggregates are suitable for use in salt environments but not for acidic environments. Crushed aggregates are preferable to rounded ones because they produce higher strength materials. Aggregates should conform to the following requirements:

2.1.2.1 *Aggregate gradation*-Graded aggregates should be used in the production of sulfur concrete to minimize binder requirements. Three size fractions of aggregate are generally used in preparing the dense-graded product:

a) coarse aggregate,
b) fine aggregate, and
c) mineral filler (minus 200 mesh, 75 μm).

The gradation should result in minimum voids in the mineral aggregate (VMA). Table 2.1.2.1, prepared in accordance with ASTM D 3515, may be used as a guide for achieving minimum voids in the mix.

2.1.2.2 *Corrosion resistance of aggregates*-Aggregates for sulfur concrete in acidic environments should not show any effervescence when tested in acid of the expected concentration and at the expected temperature of exposure. Aggregates for sulfur concrete to be used in acidic environments should also have less than 2 percent loss in weight when leached for 24 hr in acid of the expected concentration and at a temperature of 140 ± 5 F (60 ± 3 C).

Aggregates for sulfur concrete in salt environments should show no reaction or deterioration after leaching 24 hr in the expected salt solution at a temperature of 140 ± 5 F (60 ± 3 C).

2.1.2.3 *Moisture absorption of aggregates*-Porous aggregates should not be used. Instead, the aggregates should be highly impervious and highly resistant to freeze-thaw stresses. Maximum moisture absorption should be less than 1 percent for coarse aggregates and less than 2 percent for fine aggregates, as determined by using ASTM C 127 and C 128 procedures, respectively.

2.2-Mixture preparation and testing

2.2.1 *Mixture preparation*-Manufacture of durable, corrosion-resistant sulfur concrete requires good quality control of all components used in its production. Good construction practices are also necessary at both the mixing plant and the construction site. The contractor should be able to control accurately the proportions of each ingredient in the mixture, and report them.

2.2.1.1 *Properties*-Table 2.2.1.1 gives properties that are typically achieved after one day of cooling for sulfur concrete.

Table 2.1.2.1-Dense-graded aggregate gradation limits (ASTM D 3515)

Sieve size	1 in. (25 mm) aggregate, percent passing	¾ in. (19 mm) aggregate, percent passing	½ in. (12.5 mm) aggregate, percent passing	⅜ in. (9.5 mm) aggregate, percent passing
1½ in. (37.5 mm)	100			
1 in. (25 mm)	90-100	100		
¾ in. (19 mm)		90-100	100	
½ in. (12.5 mm)	56-80		90-100	100
⅜ in. (9.5 mm)		56-80		90-100
4 (4.75 mm)	29-59	35-65	44-74	55-85
8 (2.38 mm)	19-45	23-49	28-58	32-67
50 (300 μm)	5-17	5-19	5-21	7-23
200 (75 μm)	1-7	2-8	2-10	2-10

Table 2.2.1.1-Properties of sulfur concrete after one-day cooling

Compressive strength, minimum, psi (MPa)	4000 (27.6)
Flexural strength, minimum, psi (MPa)	750 (5.2)
Splitting tensile strength, minimum, psi (MPa)	600 (4.1)
Absorption after 1 day immersion in water,* maximum percent	0.10
Air voids, percent	4-8
Coefficient of thermal expansion, maximum, per deg F (C)	8.3×10^6 (15×10^6)
Freeze-thaw durability (ASTM C 666, Method A), dynamic modulus of elasticity retention after 300 cycles, minimum percent	60
Modulus of elasticity, psi (GPa)	$3\text{-}4 \times 10^6$ (20.7 – 27.6)
Aggregate gradation	Table 2.1.2.3

* Test procedure is described in Section 2.2.3.6.

2.2.2 Mixture proportions

2.2.2.1 *General*-Mixture proportions are the amounts of coarse aggregate, fine aggregate, mineral filler, and sulfur cement required to obtain a high-quality sulfur concrete mixture. In this guide, the term mixture design is used to indicate the considerations and procedures followed for determining the optimum mixture proportions; this should not be confused with structural design.

Mixture design for sulfur concrete is quite different from mixture design for portland cement concrete. Aggregates for sulfur concrete meeting the gradation requirements of ASTM D 3515 will produce more workable concrete than those complying with ASTM C 33 specifications.

However, as with portland cement concrete and asphaltic concrete, the optimum mixture design for sulfur concrete must take into consideration the properties desired for its specific use. The mixture designs described here will be for sulfur concrete materials suitable for construction of floors, foundations, tiles, sumps, side walls, and electrolytic cells for use in acid or salt environments.

The usual objective of mixture design is to prepare sulfur concrete with the following characteristics:

1. Resistance to attack by most acid and/or salt solutions
2. Minimum moisture absorption
3. Mechanical strength properties equivalent to or better than those of portland cement concrete
4. Sufficient fluidity for good workability
5. Minimal shrinkage on solidification

2.2.2.2 *Cement requirements*-The objective of mixture designs for sulfur concrete is to determine the sulfur cement quantity, in combination with specific aggregates, that provides the most desirable balance between mechanical strength, high specific gravity, low absorption, and good workability. Table 2.2.2.2(a) shows the range of sulfur cement levels for maximum sizes of dense-graded aggregate. The mixture is designed for moisture absorption less than 0.1 percent by mass. Table 2.2.2.2(b) contains data illustrating the properties obtained in developing a mixture design for sulfur concretes prepared with varying amounts of cement in combination with a minus ⅜ in. (9 mm) dense-graded quartz aggregate.

Table 2.2.2.2(a)-Range of cement levels

Maximum aggregate size, in. (mm)	Cement, percent by weight
1 (25.4)	12 to 15
¾ (19.1)	13 to 16
½ (12.7)	14 to 17
⅜ (9.5)	16 to 19

Using dense-graded aggregate and modified sulfur cement, the range of cement levels should be adjusted according to the type, size, and gradation of the aggregate. Tables 2.2.2.2(a) and 2.2.2.2(b) may be used to select approximate sulfur cement limits for mix design purposes. Actual proportions may fall slightly outside this range for certain material combinations.

2.2.2.3 *Voids*-Voids in sulfur concrete are important for two reasons: 1) They serve as stress relief sites, improving the durability of the material;[24] and 2) the presence of voids in sulfur concrete reduces the quantity of sulfur cement required to coat the mineral aggregate, thereby minimizing the cement-related shrinkage. The voids entrained on mixing the aggregate and modified sulfur cement are discrete and discontinuous, as observed under microscopic examination. Consequently, they do not increase the moisture absorption of the sulfur concrete. Generally, 4 to 8 percent of air voids are entrapped during the mixing of the sulfur concrete.

2.2.3 *Testing of properties*

2.2.3.1 *Sample preparation*-Sample specimens are cast from sulfur concrete materials in the mixing and working temperature range, 270 to 285 F (132 to 141 C), into standard ASTM molds as specified in ASTM C 31, using steel molds. The molds may be preheated to approximately 280 F (138 C) before adding the sulfur concrete. The material is compacted as the sulfur concrete is added to the mold by tamping with a heated ⅝-in. (16-mm) rod having a hemispherical tip. Samples are cast in an upright position and allowed to cool to room temperature before being removed from the molds. Before testing, samples are allowed to cool a minimum of one day at room temperature.

2.2.3.2 *Compressive strength*-Compressive strength measurements should be in accordance with ASTM C 39

Table 2.2.2.2(b)-Mixture design data for sulfur concrete using minus ⅜ in. (9 mm) dense-graded aggregate

Aggregate, percent by weight	Cement, percent by weight	Specific gravity	Voids, percent by volume	Compressive strength, psi (MPa)	Absorption, percent by weight	Workability
90.0	10.0	2.209	13.7	2960 (20.4)	1.06	relatively dry
87.5	12.5	2.297	9.7	6100 (42.1)	0.54	relatively dry
85.0	15.0	2.370	6.2	7360 (50.8)	0.07	stiff
82.5	17.5	2.372	5.5	7450 (51.4)	0.02	fluid
80.0	20.0	2.366	5.1	6530 (45.0)	0.01	soupy

Note: A mixture design should be made for each new source and gradation of aggregate used for preparing sulfur concrete.

or ASTM C 109. Specimens should generally be tested no sooner than 24 hr after they are made. However, in applications where high early strength is desirable, representative specimens may be tested within a shorter time period.

Sulfur concrete develops about 70 percent of its ultimate strength within a few hours after cooling, and usually 75 to 85 percent after 24 hr at 68 F (20 C).[24] Ultimate strength is commonly obtained after 180 days at 68 F (20 C).[24] The rate of strength development is, however, dependent on the temperature at which the material is aged. Strength gain is slower at elevated temperatures and faster at lower temperatures. Since larger masses of sulfur concrete cool more slowly, larger masses gain stren th more slowly but achieve the same ultimate strength.[19] These factors should be considered in deciding at what age sample specimens should be tested for strength.

2.2.3.3 *Flexural strength*-Flexural strength determinations are made in accordance with ASTM C 78.

2.2.3.4 *Splitting tensile strength*-Tensile strength measurements are made in accordance with ASTM c 496.

2.2.3.5 *Voids*-Void contents may be determined in two ways:

1. By determining the specific gravity of sulfur concrete using ASTM C 642. Voids are determined by calculation of the void content from the measured specific gravity and the theoretical specific gravity of the aggregate-cement mixture. A distinction between discrete and continuous voids should be stated as provided in ASTM c 642.
2. By microscopic determination on a section cut from a sulfur concrete specimen in accordance with ASTM C 457 using the linear traverse (Rosiwal) method.

2.2.3.6 *Absorption*-Sulfur concrete specimens are weighed, immersed in water at 68 F (20 C) for 24 hr, surface dried, and reweighed. Moisture absorption is calculated as follows

$$A = [(C - B)/B] \times 100$$

where

A = percent by weight moisture absorption
B = weight of dry 3 x 6 in. (76 x 152 mm) cylindrical specimen
C = weight of surface-dried specimen after immersion

2.2.3.7 *Coefficient of thermal expansion*-Linear thermal expansion determinations are made on ½ x ½ x 1 in. (13 x 13 x 25 mm) long specimens cut from 3 x 6 in. (76 x 152 mm) cylinders of sulfur concrete. The expansion is measured over the 77 to 212 F (25 to 100 C) temperature range at a constant heating rate of 5 ± 0.1 F (3 ± 0.1 C) per min. Aggregate size is limited to ⅜ in. maximum using this method.

2.2.3.8 *Freeze-thaw durability*-Freeze-thaw measurements are determined in accordance with ASTM C 666 using Procedure A, "Rapid Freezing and Thawing in Water," on 3 X 3 x 14-m (76 x 76 x 356-mm) bars of sulfur concrete. Dynamic modulus of elasticity is determined by ASTM C 215.

2.2.3.9 *Modulus of elasticity*-Modulus of elasticity determinations are made on 3 x 6 in. (76 x 152-mm) cylinders of sulfur concrete in accordance with ASTM C 469.

2.2.3.10 *Aggregate gradation*-Aggregate gradation determinations are made on 3 x 6-in. (76 x 152-mm) cylinders of sulfur concrete, after burning off the cement in a muffle furnace. Initial combustion is at 302 F (150 C). When the bulk of the cement has been removed by combustion, the temperature is raised to 825 F (440 C) and held until constant weight is achieved. After cooling to ambient temperature, the gradation of the aggregate is determined in accordance with ASTM C 136.

In addition to determining the aggregate gradation, the cement content of the concrete may be estimated from the loss in weight on burning off the cement from the sulfur concrete.

2.2.3.11 *Swelling clays in sulfur concrete*-Sulfur concrete should not contain swelling clays because these clays can cause premature failures. An effective test for swelling clay is to immerse a 6 x 6 x 1 in. (152 x 152 x 25 mm) sample of sulfur concrete in water. The sample is first weighed dry then dipped in water, towel dried, and weighed again. The sample is then immersed in hot water of 180 F (82 C) minimum for 24 hr, removed, examined, towel dried, and weighed. Following this, the sample is reimmersed in the hot water. Removal, examination, towel drying, weighing, and reimmersion are repeated daily until failure is observed.

The first sign of failure is usually a gain in mass (or "weight") of 1 percent or more. This will usually occur sometime between the second and fourth day if swelling clay is present. The weight gain will soon be followed by spalling, hairline cracks, and additional weight gain of 3 to 5 percent. If large amounts of swelling clays are present, cracking may be so extensive that the sample cannot be recovered from the hot water.

2.3--Preparation of subbase or surface to receive sulfur concrete

The most important requirement of a subbase or contact surface is dryness. When the subbase is relatively free from moisture, a 2 to 4-in. (51 to 102-mm) layer of dry sand may be used to bring the subbase to grade. Whenever moisture is present, a 6-mil polyethylene or similar vapor barrier should be installed over the sand prior to sulfur concrete placement to reduce vaporization of water while placing the hot concrete mixture. Regardless of the type of base material used, the base should be well consolidated or compacted.

Whenever sulfur concrete is installed over existing portland cement concrete, deteriorated areas should be removed to assure that a sound base is provided, and the existing concrete should be dried as much as is practical. Use of a vapor barrier or other flexible membrane to prevent moisture infiltration should also be considered.

2.4-Batching, transporting, placing, and finishing

2.4.1 *Equipment requirements*-Sulfur concrete is produced by mixing heated aggregate (350 to 400 F [177 to 204 C]) with modified sulfur cement and fine mineral filler (ambient temperature) to prepare a uniform, well-mixed concrete that is then maintained within a temperature range of 270 to 285 F (132 to 141 C) until placed. The heated aggregates melt the sulfur cement and heat the fine mineral filler.

The minimum and maximum temperatures of a sulfur concrete mixture are controlled because: a) the modified sulfur cement melts at 246 F (119 C) and b) above 300 F (149 C), the viscosity of sulfur concrete rapidly increases to an unworkable consistency. For these reasons, 270 to ***285*** F (132 to 141 C) has been found to be an optimum temperature range to allow time for transportation, placement, and finishing of the sulfur concrete before solidification.

Equipment and techniques from both the concrete and asphalt technologies are used to batch, transport, place, and finish sulfur concrete. The typical equipment for a cast-in-place sulfur concrete operation would include the following:

1. Aggregate drying system
2. Weigh hopper/scale for proportioning mixture materials
3. Mixing/transporting equipment
4. Typical concrete hand-placing and finishing tools

2.4.2 *Aggregate drying/heating equipment*-An efficient means of drying and heating aggregates are rotary kiln systems (manufactured for the asphalt industry). Specially heated concrete transit mixer trucks can be modified for preparation of sulfur concrete.

Sand and coarse aggregates are typically loaded into separate bins and fed cold through feed gates that proportion the correct amount of sand to rock. After passing through the gates into the rotary kiln, the sand and rock are mixed and superheated by either a propane or diesel-fueled burner to remove the moisture and to raise the aggregate temperature above the melting point of sulfur. On some systems, an exhaust and stack system vents combusted gases and collects fines mixed with the exhaust. These fines are then returned to the mixed and heated aggregates at the discharge end of the kiln. After discharge from the kiln, heated aggregates are typically loaded into the mixer/transport equipment via some type of bucket-and-chain or conveyor system. As an intermediate step before loading the aggregates into the mixer/transport equipment, hot aggregates can be discharged into a weigh hopper/scale system for accurate weighing.

Several quality control measures should be observed throughout the batching process:

1. Stockpile maintenance to avoid contamination of aggregates.
2. Verify aggregates on-site for gradation, moisture absorption, acid resistance, and absence of swelling clays.
3. Calibrate of material-weighing equipment.
4. Sample batched sulfur concrete to test for compressive strength, moisture absorption, density, etc.

Aggregates should be heated so that the final sulfur concrete mixture is 270 to 285 F (132 to 141 C). Generally, whenever solid sulfur cement is used, the aggregate should be heated to 350 to 400 F (177 to 204 C) to obtain a final aggregate-liquid sulfur-filler mixture temperature at 270 to 285 F (132 to 141 C).

2.4.3 *Mixer/transport equipment*-The requirements for mixing/transporting equipment are defined by the unique thermoplastic characteristics of sulfur concrete:

1. Sulfur concrete must be maintained in molten form within a narrow temperature range.
2. The concrete mixture must be thoroughly mixed so that the molten sulfur cement adequately coats the fine and coarse aggregate and mineral filler. There should be minimal segregation of aggregates in the mixture.

Special consideration should be given to several items to insure good quality control:

1. Material should be batched in the following sequence:
   a. Heated sand and coarse aggregate
   b. Modified sulfur cement or sulfur, then concentrate
   c. Fine mineral filler, fly ash (Type F), or silica flour (adding the filler after the sulfur prevents problems from dusting and balling of the filler)
   d. Fibers (if used)
2. The temperature of the mixture should be continuously monitored to prevent under-or overheating. By frequently monitoring the mixture temperature and adjusting the propane burner feed as required, the mixture can be kept within a narrow temperature range prior to placement, avoiding the problems of underheating previously mentioned.
3. Sulfur concrete has a stiff, dry appearance when it is heated above ,300 F (149 C), and the contractor should not attempt to enrich the mix with additional sulfur cement. When this happens, the temperature should be checked to determine whether the mixture is too hot. If it is, the mixture temperature should be quickly reduced below 300 F (149 C). If the right amount of sulfur cement was originally added, the sulfur concrete will return to a more fluid and workable consistency.

2.4.4 *Forming and reinforcement*-Both wood and metal forms can be used. Petroleum-based form release agents should be used on wall pours but are not necessary on slab forms. When forming large surface areas (i.e., walls) with reusable metal forms, they should be preheated to prevent formation of a skin of sulfur cement caused by flash setting of the sulfur concrete when it contacts a cold form.

Sulfur concrete can be reinforced with Grade 60 reinforcing steel (ASTM A 615, A 616, A 617, and A 706) (modified sulfur does not react with steel), epoxy-coated reinforcing steel (ASTM A 775), or with glass fibers. Standard details for steel in portland cement concrete is recommended for design with sulfur concrete. Clearances between forms and reinforcing steel should be increased slightly to avoid placement difficulties due to freezing of sulfur concrete on reinforcing steel. An alternative to increased clearance is preheating of the forms and reinforcing steel using indirect heat such as infrared heaters, prior to placement of sulfur concrete.

Glass fibers have been very effective as an enhancement in field applications for controlling shrinkage cracks and improving the ductility and impact resistance of sulfur concrete. When using glass fibers, approximately 15 to 20 lb. (6.8 to 9.1 kg) of 0.5 to 1.5 in. (13 to 38 mm) long fibers can be added to every cubic yard (0.8 m) of sulfur concrete. Further information may be obtained from the U.S. Bureau of Mines Report of Investigations, No. 8956.[53]

2.4.5 *Placing and finishing*

2.4.5.1 *General*-The keys to successful placing and finishing of sulfur concrete are: (a) having the sulfur concrete mass heated to 270 to 285 F (132 to 141 C) at the moment of placement; and (b) speed in placing and finishing.

Concrete buggies can be used to transport the hot sulfur concrete to the forms. While these buggies can be insulated to reduce the heat loss from the sulfur concrete, it is normally not necessary. The buggies should always be as full as can be safely handled and the workers should discharge their buggy loads quickly. Sulfur concrete should be placed as rapidly as possible so that strikeoff and finishing can be accomplished while the concrete is hot enough.

2.4.5.2 *Floor construction*-Compaction, strikeoff, and finishing of sulfur concrete can be done with conventional portland cement concrete hand tools. Sulfur concrete should be worked in as large a mass as possible to maintain heat. The maximum dimensions for slabs are normally limited by the ability of the finishing crew to finish the concrete while it is sufficiently hot.

Once the placement section is filled with sulfur concrete it should be struck off with a simple screed. With a properly designed mixture, probe vibrators are generally not necessary. A vibrating screed is effective in achieving a relatively smooth, sealed finish.

Once the slab is struck off, there are only a few minutes available to finish the surface (depending upon ambient conditions). When placing a 2-in. (51-mm) overlay, possibly 2 to 10 min are available to make one finishing pass before the surface will begin to crust and solidify. With a 4 to 8-m slab (102 to 203 mm), 5 to 20 min are available for finishing. Finishing can be accomplished using conventional construction crews and techniques.

High-quality wood and metal floats are recommended for the finishing. One pass over the slab surface while it is still molten to smooth and seal the surface is all that is required. If the finisher continues to work the surface as it starts to crust, the crust will tear and the finish will be destroyed. If this happens, a propane torch can be used to quickly remelt that area so that it can be refinished.

While finishing, the sulfur concrete will stick to the floats and build up a layer on them. The finisher should keep a bucket of cold water close by so that the float can frequently be immersed in water to flash-set the sulfur concrete sticking to the tool. The float can then be rapped sharply against a hard surface to pop the sulfur concrete off the tool.

When sulfur concrete is unintentionally spilled, it should be left in a mass to solidify. No attempt should be made to quickly shovel and scrape it up because this will leave a thin film bonded to the surface, which is difficult to remove. A solidified mass of sulfur concrete can be removed more easily with a crowbar or shovel.

Sulfur concrete's final (as screeded) finished texture and skid resistance are generally desirable in most industrial environments. The surface of the slab is dense, washes down easily, and provides good abrasion resistance.

2.4.5.3 *Wall constmction*-Due to some of the unique characteristics of sulfur concrete, wall construction should be given some special considerations. The difference in temperature between sulfur concrete and the reinforcing steel and forms should be considered. Preheating the reinforcing steel and forms using infrared or other suitable heaters is usually necessary to preclude poor consolidation due to flash-setting of sulfur concrete. The use of insulation on the outside of wall forms to retain heat during placement of sulfur concrete should also be considered. Insulation will provide additional time at working temperatures to assure adequate consolidation of successive lifts, resulting in a monolithic wall. A thin coating of release agent on the wall forms is necessary to permit easy removal of the forms after the sulfur concrete has cooled. Internal vibration (light) may be used to insure consolidation. Excessive vibration can result in segregation. External vibration has been used effectively for consolidation.

2.4.5.4 *Placing on steep grades*-Earlier experiences with sulfur concrete demonstrated that there was a limit to the grade at which sulfur concrete could be placed without excessive flowing of the sulfur concrete to the low side. Recent experience has shown that sulfur concretes that contain angular aggregates can be placed without this flowing problem on grades ranging from ¼ in. per ft (2.1 percent) to as steep as ¾ in. per ft (6.5 percent) by controlling two mixture characteristics:

1. Sulfur concrete using silica flour as mineral filler can be placed on steeper grades than sulfur concretes using fly ash as mineral filler. Mixes with silica flour are

generally stiffer and flow less on sloping surfaces.

2. The excessive flowing problem can-be minimized by designing mixtures with sulfur cement contents at the minimum design levels.

2.4.6 *Repair of damaged surfaces*-Sulfur concrete surfaces that were not properly finished or that have become damaged in use can be reheated and refinished. The area can be remelted by using indirect heating, such as with an infrared heater, and then quickly refinished or repaired as needed. It is not advisable to use a concentrated source of heat, because concentrated heat on a cold sulfur concrete slab can induce stress cracks on thin sections.

Holes, spalls, voids, and other minor surface blemishes can be patched easily by reheating the damaged area and filling with sulfur mortar.

2.5-Joints and joint sealing

Joints are formed to control cracking, terminate placements, and allow for thermal expansion and contraction. Location of joints should be evaluated on a job-to-job basis after consideration of the placing sequence. Normally, the width of placements is limited to 8 to 12 ft. (2.2 to 3.7 m) because of the limitation of finishing.

Typically, contraction joints should be constructed at a distance in feet of not more than three times the slab thickness in inches. Contraction joints are usually ¼ in. (6 mm) to ¾ in. (9 mm) wide and extend 20 to 25 percent into the depth of the slab. If a construction joint is formed at the termination of a placement, the incorporation of a keyway is beneficial but not normally necessary.

Expansion joints are usually ¾ to ½ in. (10 to 13 mm) wide. They are constructed to relieve thermal stresses and to separate slabs from the vertical surfaces of structural members. Maximum spacing of expansion joints should be limited to 60 ft. (18 m) for a normal design mixture. If higher sulfur content is used (greater than 18 percent by weight), the maximum distance should be reduced accordingly. Preformed expansion joint filler made of asphalt-saturated fiberboard meeting ASTM D 1751 has performed satisfactorily.

A flexible joint sealant compatible with the environmental conditions and ACI 504R-77, "Guide to Joint Sealants for Concrete Structures," should be applied to all joints, extending the full width of the joint with a depth no greater than the width. A bond breaker should be provided below the joint sealant. A preformed backer rod made of closed-cell polyethylene foam is often used as a bond breaker and will also keep the uncured sealant from seeping through the joint.

CHAPTER 3--REFERENCES

3.1-Specified and/or recommended references

The documents of the various standards-producing organizations referred to in this document are listed below with their serial designations.

American Concrete Institute

504R Guide to Joint Sealants for Concrete Structures

ASTM

A 615 Standard Specification for Deformed and Plain Billet-Steel Bars for Concrete Reinforcement

A 616 Standard Specification for Rail-Steel Deformed and Plain Bars for Concrete Reinforcement

A 617 Standard Specification for Axle-Steel Deformed and Plain Bars for Concrete Reinforcement

A 706 Standard Specification for Low-Alloy Steel Deformed Bars for Concrete Reinforcement

A 775 Standard Specification for Epoxy-Coated Reinforcing Steel Bars

C 31 Standard Practice for Making and Curing Concrete Test Specimens in the Field

C 33 Standard Specification for Concrete Aggregates

C 39 Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens

C 78 Standard Test Method for Flexural Strength of Concrete (Using Simple Beam with Third-Point Loading)

C109 Standard Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2 in. or 50-mm Cube Specimens)

C 127 Standard Test Method for Specific Gravity and Absorption of Coarse Aggregate

C 128 Standard Test Method for Specific Gravity and Absorption of Fine Aggregate

C 136 Standard Method for Sieve Analysis of Fine and Coarse Aggregates

C 215 Standard Test Method for Fundamental Transverse, Longitudinal, and Torsional Frequencies of Concrete Specimens

C 457 Standard Practice for Microscopical Determination of Air-Void System in Hardened Concrete

C 469 Standard Test Method for Static Modulus of Elasticity and Poisson's Ratio of Concrete in Compression

C 496 Standard Test Method for Splitting Tensile Strength of Cylindrical Concrete Specimens

C642 Standard Test Method for Specific Gravity, Absorption, and Voids in Hardened Concrete

C 666 Standard Test Method for Resistance of Concrete to Rapid Freezing and Thawing

D 70 Standard Test Method for Specific Gravity and Density of Semi-Solid Bituminous Materials

D 1751 Standard Specification for Preformed Expansion Joint Filler for Concrete Paving and Structural Construction (Nonextruding and Resilient Bituminous Types)

These publications may be obtained from the following organizations:

American Concrete Institute
P.O. Box 19150
Detroit, MI 48219-0150

ASTM
1916 Race Street
Philadelphia, PA 19103

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