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Sulfur mortar and polymer modified sulfur mortar lining for concrete sewer pipe

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Sulfur mortar and polymer modified sulfur mortar lining for concrete sewer pipe

by

Thaer M. Wahshat

A dissertation submitted to the graduate faculty
in partial fulfillment of the requirements for the degree of
DOCTOR OF PHILOSOPHY

Major: Civil Engineering (Geotechnical Engineering)

Program of Study Committee:
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For the Major Program

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INTRODUCTION

It is well known that most of the modern sewer pipes in the world are made of reinforced Portland cement concrete. Reinforced concrete has many advantages such as its high strength characteristics, durability, versatility, and low cost when used as a construction material. Many other materials have been used to manufacture sewer pipes such as PVC, vitrified clay, and ductile iron. However, reinforced concrete pipe remains the most popular type among all because of the characteristics listed above.

An existing problem that faces concrete sewer pipe is corrosion by sulfuric acid. A good portion of the concrete sewer pipes in the United States have been attacked by sulfuric acid (Pitt, 1995) as a result of biogenic activities or by direct oxidation of hydrogen sulfide produced in sewage.

It is estimated that in the district of Los Angeles County alone, 208 km of a total of 1900-km concrete sewer lines have been subjected to significant corrosion (Sydney et al. 1996). The cost of replacing or rehabilitating the deteriorated pipe has been estimated at $396 million. According to the EPA, the total estimated length in need of rehabilitation is approximately 800,000 miles of sanitary sewer lines in the US (Pitt, 1995). The severity of the problem emphasizes the fact that new sewage concrete pipes must be protected and older ones must be rehabilitated.

Undesirable experience with concrete pipe is leading to use of other alternative pipe materials including fiberglass, plastic and plastic lined concrete pipe. Although this appears to be a technically suitable solution, plastic increases the cost of the pipe by three to five times. In addition, these solutions do not facilitate the versatile size ranges that concrete sewage pipe can offer. In addition, the concrete pipe industry is losing a good portion of its current market; it is projected that 25% of the concrete sewer pipe industry will be lost by the year 2003 to the other types of pipe.

Because of its unique chemical and physical properties, elemental sulfur has interesting potential applications for protecting concrete pipe, particularly in the construction industry (Schmidt, 1977). Despite years of research and development activities, little sulfur has been consumed in these applications. One reason for this is that sulfur atoms combine with each other to form the extremely complicated and complex system of chain or ring molecules, S_x. Depending on x, the physical and chemical properties of sulfur and molecular equilibria mixtures change drastically.

Elemental sulfur was proposed as a protective coating for concrete in foundations, dams, sea walls, catch basins and other structures which are subjected to corrosion as early as 1930 (Fike, 1976). However, very little was done in application for many years because of technical problems and the high cost of sulfur. Elemental sulfur could not form a continuous barrier because of volumetric instability from changes in molecular structure after solidifying and it was relatively expensive since sulfur had to be mined.

Recent work sponsored by U. S. Bureau of Mines solved the technical problem by developing modifiers that stabilize the solid structure of sulfur. A secondary effect of the Clean Air Act solved the problem of cost and availability by mandating sulfur removal from refined petroleum products. There is now an abundance of elemental sulfur at many locations throughout the country.

The proposed method of manufacturing acid resistant pipe involves casting linings of a special formulation of sulfur cement mortar. This method has the advantage of creating an exceptionally durable product that costs a fraction of amount for the other acid resistant alternatives, such as, plastic or plastic lined pipe.

RESEARCH OBJECTIVES

The intent of this research is to evaluate and line concrete sewer pipe using a low cost acid resistant material; i.e. sulfur and modified sulfur mortars. Also, to determine the technical and engineering properties of the liner and the steps involved in the development process. Specific objectives are as follows:

- Determine strength and time characteristics of sulfur and DCP modified sulfur mortars;
- Determine the effect of aggregate content on strength properties of the sulfur and DCP modified sulfur mortar;
- Determine viscosity characteristics of the sulfur and modified sulfur mortars;
- Determine the bond strength between modified and unmodified sulfur mortar and concrete substrate under application conditions;
- Determine shrinkage characteristics of the modified and unmodified sulfur mortar;
- Determine the optimum content of sulfur modifier, Dicyclopentadiene (DCP) to obtain the best application properties;
- Select a pipe sample that represents typical sewer pipes used in Iowa to use as a prototype for the lining process;
- Determine the optimum joint configuration that meets the market requirements and apply it in the lining process;
- Design a system that will enable the lining of concrete pipes with sulfur mortar without affecting the pipe manufacturer's process of manufacturing the pipe; and
- Produce a full-scale lined concrete sewer pipe.

THEORETICAL BACKGROUND

The theoretical background summarizes previous research related to the hydration of Portland cement concrete, sulfuric acid derivation in sewer pipes, mechanism of sulfuric acid corrosion of concrete sewer pipe, properties of elemental sulfur and sulfur mortar, and polymer sulfur modification and properties of modified sulfur.

Portland Cement Concrete and Concrete Pipe

Hydration of Portland Cement

Cement is a complex chemical compound that is made up of four major compounds: Tricalcium Silicate (C_3S), Dicalcium Silicate (C_2S), Tricalcium Aluminate (C_3A), and Tetracalcium Aluminoferrite (C_4AF). Table 1 gives a typical composition of Type I Portland cement with the expected percentage of each compound.

When water is added to Portland cement, the basic compounds present are transformed to new compounds by chemical reaction (hydration) according to the following reactions:

C_3S + water→ CSH (tobermorite gel) + $Ca(OH)_2$

C_2S + water→ CSH (tobermorite gel) + $Ca(OH)_2$

C_4AF + water + $Ca(OH)_2$ → Calcium Aluminoferrite Hydrate

C_3A + water + $Ca(OH)_2$ → Tetracalcium Aluminate Hydrate, and

C_3A + water + Gypsum → Calcium Monosulfoaluminate

Of the product, 25% by weight is calcium hydroxide and 50% by weight is cement gel (CSH) or tobermorite gel that is the major bonding material in the cement gel. The chemical composition of tobermorite gel is $CaO.SiO_2.H_2O$.

Table 1: Typical basic composition of type I Portland cement

Compound	Formula	Composition (%)	Shorthand Notation
Tricalcium silicate	$3CaO.SiO_2$	42-67	C_3S
Dicalcium silicate	$2CaO.SiO_2$	8-31	C_2S
Tricalcium aluminate	$3CaO.Al_2O_3$	5-14	C_3A
Tetracalcium aluminoferrite	$4CaO.Al_2O_3.Fe_2O_3$	6-12	C_4AF
Calcium sulfate dihydrate	$CaSO_4.H_2O$	2.6-3.4	CSH_2
Free lime	CaO	0-1.5	C
Magnesium oxide	MgO	0.7-3.8	M
Volatiles	—	0.6-2.3	—

Concrete Sewer Pipe

Concrete pipes are known to have been used by the Romans for water supply and other applications since the second century AD (Perkins, 1974). Some of the concrete pipes found in Cologne, Germany, were in reasonable shape and a functional condition even after more than 1800 years. This kind of behavior and durability is rare and exclusive to only a few materials.

The first concrete pipe was laid in the US around 1842, and 1875 in Britain. The first concrete sewer pipe in the US was laid in Mohawk in New York.

In 1912 the first ASTM committee was formed in order to protect the quality, uniformity, performance and specifications of the concrete sewer pipe (Loving, 1938). The committee was designated as ASTM Committee C-4. The first standard specifications were introduced in 1920. In 1935 ASTM C14-35: Standard Specification for Concrete Sewer Pipe, and ASTM C75-35: Standard Specifications for Reinforced Concrete Sewer Pipe were adopted.

Sulfuric Acid Reaction with Concrete

Concrete is susceptible to corrosion by strong mineral acids such as sulfuric and hydrochloric acids (Raju and Dayaratnam, 1984). Unlike other acids, sulfuric acid will cause both dissolving and swelling of concrete. Several researchers reported loss of weight of concrete when exposed to percentages of sulfuric acid (Pietrzykowsky, and DePuy, 1975).

When sulfuric acid is present with concrete, sulfuric acid (H_2SO_4) reacts with the calcium hydroxide ($Ca(OH)_2$) and cement gel (CSH) in concrete to form calcium sulfate ($CaSO_4$) (Attigobe and Rizkalla, 1988). The reaction equations are as following:

$H_2SO_4 + Ca(OH)_2 \rightarrow CaSO_4 + 2H_2O$ and

$H_2SO_4 + CSH \rightarrow CaSO_4 + SiO_2 + 2H_2O$

The reaction results in a type of deterioration that is called gypsum corrosion and causes the dissolution and swelling of concrete. When gypsum corrosion occurs, the alkalinity of the liquid in concrete is reduced, which is favorable to microbiologically influenced corrosion. The product of corrosion is a weak compound with no cementitious properties and the active formation eventually leads to the destruction and collapse of the concrete pipe.

Several alternatives were studied by the engineering and chemical communities to improve concrete properties against sulfuric acid attack or to inhibit the production of corrosive materials in the environment. Research showed that long term exposure test data showed that the partial replacement of Portland cement by fly ash and silica fume can effectively improve the resistance of the mortar to sulfuric acid and sulfate solution attack, although, the degree of resistance varies depending on the solution type (Torii et al. 1994).

Other alternatives included the control of the Thiobacillus organisms in order to inhibit the production of sulfuric acid. Corrosion of the concrete sewer pipes is associated with the biological oxidation of hydrogen sulfide to sulfuric acid by bacteria of the genus Thiobacillus (Padival et al. 1995). The process of bacteria inhibition under a controlled environment involved the introduction a competitor organism (a heterotrophic competitor) that reduced the acid formation by 85%.

Most concrete produced in the industry is manufactured by mixing carbonaceous aggregate with the cement paste. Similar to cement paste, carbonaceous aggregate is susceptible to acid attack; therefore, the use of non-acid-reactive aggregate was an essential part of the process of modification of the concrete pipe to become sulfuric acid resistant. Silica based aggregate was used because it is not reactive with sulfuric acid.

Acid formation in sewer pipes

Figure 1 presents of the sulfur cycle (Kienow, 1989). This Figure presents some of the biological transformations that are involved with sulfur and sulfuric compounds, and specific emphasis is given to the reactions involving hydrogen sulfide because they define what occurs in a sewer pipe. This involves the principal biological transformations that are involved starting with SO^{-4} including reduction, immobilization, mineralization, and oxidation. Other types of oxidation include chemical oxidation of sulfur where the sulfide is oxidized with oxygen, chlorine, or hydrogen peroxide. The following reactions present the generation of sulfur or hydrogen sulfide from organic matter:

$2CH_3CHOH + SO_2^{-4} \rightarrow 2CH_3COOH + S^{-2} + 2H_2O + CO_2$

$4H_2 + SO_4^{-2} \rightarrow S\text{-}^{2} + 4H_2O$ (Bacterial Reduction)

$CH_3SCH_2CHNH_2COOH + H_2O \rightarrow CH_3SH + NH_3 + CH_3CH_2COCOOH$

$CH_3SH + H_2O \rightarrow CH_4OH + H_2S$ (Mineralization)

$Cl_2 + H_2S \rightarrow 2H^+ + 2Cl^- + S^0$

$4Cl_2 + H_2S + H_2O \rightarrow 10H^+ + 18Cl^- + SO_2^{-4}$ (Oxidation with Chlorine)

Sewer gas consists primarily of hydrogen sulfide created by decomposition of organic matter (Young, 1990). Hydrogen sulfide combines with oxygen and water condensed on concrete walls in general, leading to the formation of sulfuric acid.

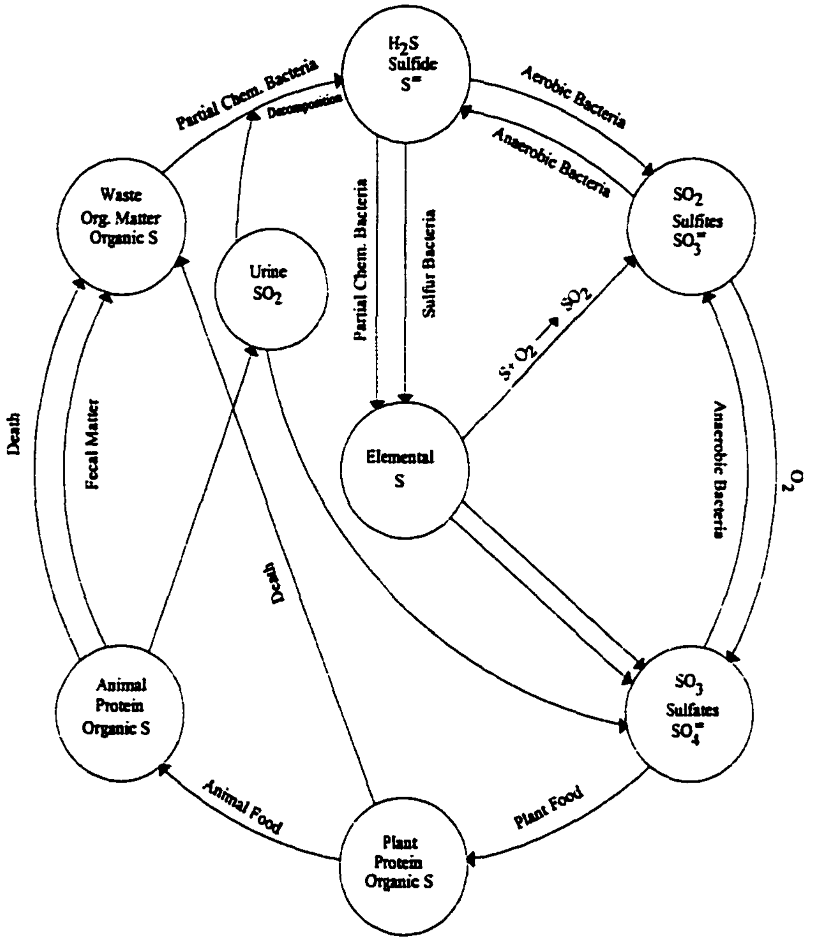


Figure 1: Sulfur cycle in the environment (Kienow, 1989)

The sulfuric acid that causes corrosion of sewer pipeline is generated by a complex ecosystem, which has been studied (Thornton, 1978). The interactions of the environment (temperature, humidity, pH), the sewer stream (flow rate, slope, ventilation, chemical and biological makeup), and the composition of the concrete used in manufacturing the pipe control this ecosystem.

When the pipe is placed into service, a succession of bacteria, each of which flourishes at certain pH levels, begins to grow. As a result, each successive species or generation of bacteria systematically lowers the pH of the concrete surface that will ultimately lead to concrete corrosion.

The biogenic corrosion phenomenon is a two-phase process (Sydney et al., 1996). In the first phase, sulfate-reducing bacteria reduces sulfate in the wastewater to sulfide when the wastewater stream is anaerobic. That is:

$$SO_4^{2-} + \text{ORGANIC MATTER} \xrightarrow{\text{Bacteria}} S^{2-} + \text{various alcohol and organic acids.}$$

Sulfates are normally present in the domestic waste supply and may be greatly increased by industrial waste and laundry detergents. Sulfide in the liquid phase coexists as dissolved H_2S, HS^- and S^{-2}. At the liquid-gas interface, dissolved H_2S is vaporized from the liquid phase into the gaseous phase.

In the second phase, some of the H_2S in the sewer headspace dissolves in the moisture present on the crown, and then sulfide is either oxidized to sulfuric acid by the sulfide-oxidizing bacteria or by direct oxidization. Over time, the pH in the sewer line head space changes from alkaline to acidic. Microscopic organisms living in the crown have been shown to lower the pH of fluid on the concrete surface to values between 1 and 3 standard units (Sand et al. 1987). Thus the attack of sulfuric acid on concrete occurs. The processes occurring in sewers with and without sulfide buildup are illustrated in Figures 2 and 3 respectively (Mclaren, 1984).

Corrosion and hydrogen sulfide generation in wastewater sewers are dependent upon the occurrence of the relevant conditions that prosper and lead to the generation of sulfuric acid including the presence of dissolved sulfides, lower pH, BOD, increased temperature, dissolved oxygen, wastewater velocity, surface junctions and sewer conditions of ventilation.

Acid production is also dominated by several species of the genus Thiobacillus (Islander et al. 1991). The sulfide-oxidizing bacteria found on sewer crowns prosper at low pHs, which are inhibitory to most competitors. The ecosystem is hostile for most organisms and is dominated by a single energy source, sulfide. The surviving microorganisms become very abundant. However, at the end, all microbiological activity comes to a halt because the environment becomes acidic to the extent that even the most resistant species of Thiobacillus can not exist.

In countries that are characterized by high air temperature and humidity, the process of the corrosion was intensified due to fact that an ideal environment for the anaerobic bacterial activity and hydrogen sulfide was created (Saricimen, 1987). Severe deterioration was noticed on the unsubmerged concrete portion of concrete sewer pipe.

An obvious solution to the problem of acid attack is to use acid resistant materials. The use of vitrified clay pipe was one of the solutions, but its disadvantage is the fact that the clay pipe cannot be reinforced. Thus diameters are limited and seals at joints have reportedly been problematic. The modern solution has been plastic liners for large diameter pipe and the use of all plastic for smaller diameter pipe. The process herein uses a liner for concrete pipe, which offers the advantages of size versatility and lower cost when compared to plastic pipe.

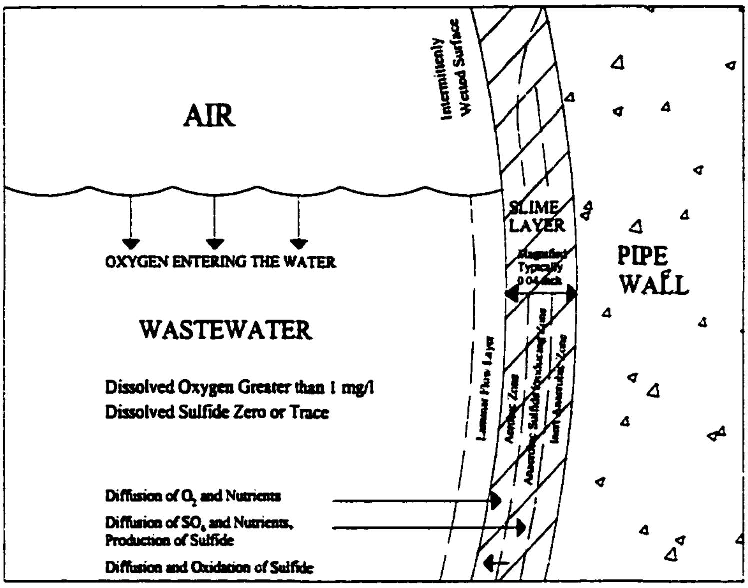


Figure 2: Processes occurring in sewers with sufficient oxygen to prevent sulfide from entering stream (Mclaren, 1984)

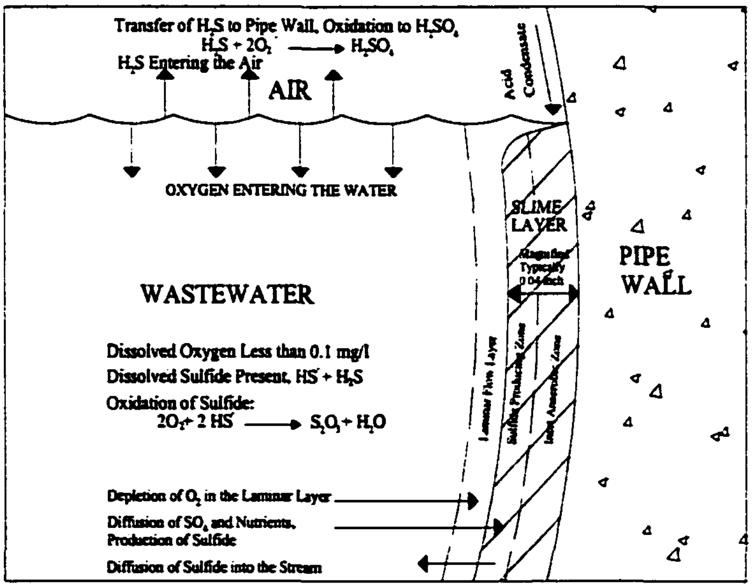


Figure 3: Processes occurring in sewers under sulfide buildup conditions (Mclaren, 1984)

Sulfur

Elemental Sulfur

Sulfur has been known to man for thousands of years; the first reference to it being identified in the Old Testament. Genesis XIX, 24. States: " Then the Lord caused to rain upon Sodom and upon Gomorrah brimstone (sulfur) and fire from the Lord out of heaven." (Meyer, 1977).

At room temperature, sulfur can exist in at least twelve different forms (allotropes) of which, three are of practical interest: Orthorhombic sulfur is the most stable form at room temperature; polymeric sulfur is formed by quenching hot liquid sulfur; and monoclinic sulfur that forms at a temperature greater than 205°F from orthorhombic sulfur and it is the stable form of sulfur at the melting point.

Pure elemental sulfur is an odorless bright yellow solid at room temperature. Table 2 shows the mechanical properties of sulfur, and these were obtained using ASTM tests designed for Portland cement concrete. Table 3 shows the structural data for nine sulfur allotropes. Table 4 shows the physical properties of elemental sulfur (Sander et al. 1984).

The electron configuration of the sulfur atom is shown in Figure 4. The sulfur atom has six outer electrons, with two unpaired 3p electrons, thus, intermolecular bonding in this element is covalent. The resulting chains are ordered in a non-planar zigzag pattern. The two additional pair of electrons prevents free rotation around the sulfur-sulfur bond (Schmidt, 1977).

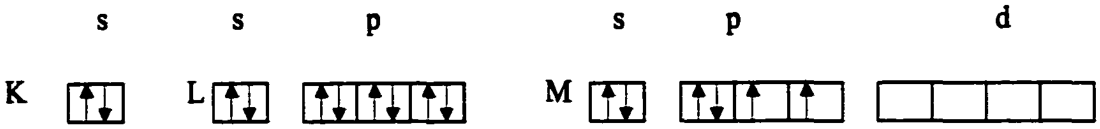


Figure 4: Electronic configuration of the sulfur atom (Schmidt, 1977)

Table 2: Mechanical properties of sulfur, results obtained using ASTM tests for concrete (Meyer, 1977)

Property	Value (psi)
Tensile strength (cast)	160
(quenched)	49-620
Compressive strength	3300
Modulus of rupture	200

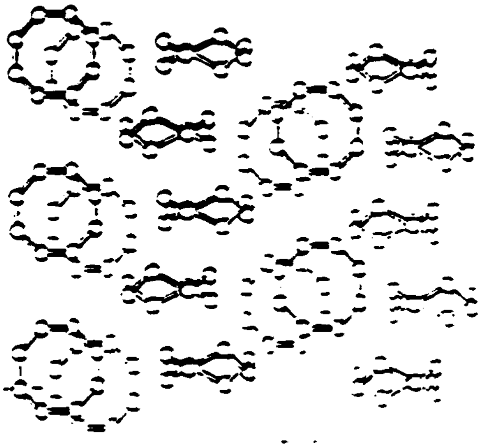
Figure 5 shows the structure of orthorhombic and monoclinic sulfur (Meyer, 1977). The orthorhombic and monoclinic contain crown shaped S_8 molecules.

When orthorhombic sulfur is heated, it first melts to a pale yellow liquid of low viscosity at about 235 °F. This liquid mainly consists of S_8 rings and it shows the expected properties of the material up to 318°F. At temperatures greater than 318°F there is a very large increase in the viscosity, followed by a gradual decrease at higher temperature as shown in Figure 6 (Tobolsky, 1965).

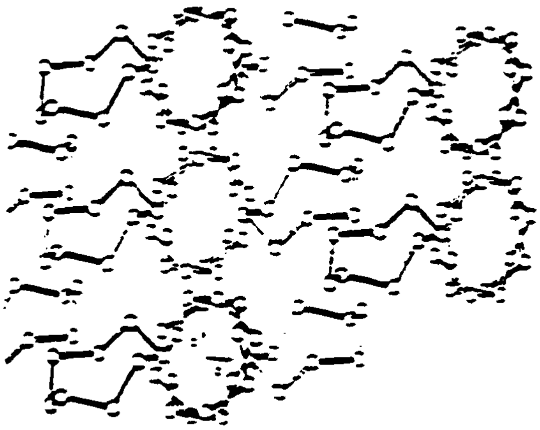
Table 3: Structure of sulfur allotropes, (Tobolsky, 1965)

Molecule	Density (g/cm3)	Melting point (°C)	Unit cell (A°)			Angle β, (degree)	Space cell
			A	B	C		
S_6	2.209	50-60	10.818	0.396	4.280±0.001	_	3;18
S_7	2.090	39	21.770	20.970	6.090	_	16;112
$S_{8\text{-}\alpha}$	2.069	94(112)	10.465	12.866	24.486	_	16;128
$S_{8\text{-}\beta}$	1.940	133	10.778	10.844	10.924	95.80	6;48
$S_{8\text{-}\gamma}$	2.198	20	8.442	13.025	9.356	124.98	4;32
S_{12}	2.036	148	4.730	9.104	14.574	_	2;24
S_{18}	2.090	128	21.152	11.441	7.581	_	4;72
S_{20}	2.016	124-125	18.580	13.181	8.600	_	4;80
S_∞	2.01	104	13.800	4x8.10	9.250	85.3	160
S_8O	2.13	20-78	19.197	7.973	8.096	_	4;32
S_7TeO_2	2.65	_	8.820	9.010	13.280	_	4;28

Orthorhombic Sulfur



Monoclinic Beta-Sulfur



Monoclinic Gamma-Sulfur

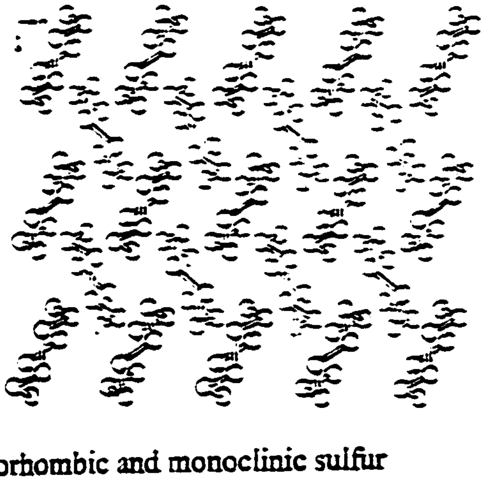


Figure 5: The structure of orthorhombic and monoclinic sulfur

Table 4: Physical properties of elemental sulfur, (Tobolsky, 1965)

Melting point	Purity 99.9% by weight of sulfur, °F	Industrial Product, °F
Orthorhombic, S_α	220	215
Monoclinic, S_β	232	223
Boiling point	772	734
Density of solid at 293 °F, g/ml		
Orthorhombic, S_α	2.07	
Monoclinic, S_β	1.96	
Amorphous, S_ω	1.92	
Density of liquid, g/ml		
240°F	1.7988	
248°F	1.7947	
265°F	1.7865	
302°F	1.7784	
Surface tension, dyne/cm		
232 °F	60.83	
282 °F	57.67	
290°F	55.00	
302°F	1.78	
Specific heat (Cp), J/mol K (T-temperature, °F)		
Monoclinic, 24.5-228 °F	$Cp=(3.58+62.4x10^{-3}T)x4.1868$	
Liquid:	$Cp=(5.4+5x10^{-3}T)x4.1868$	
Heat of fusion, J/g		
Orthorhombic, 218 °F	49.82	
Monoclinic, 230 °F	38.52	
Dynamic viscosity of liquid, Pa.s		
232 °F	0.0015	
265 °F	0.0080	
295 °F	0.0064	
298°F	5.9200	
302°F	86.3040	

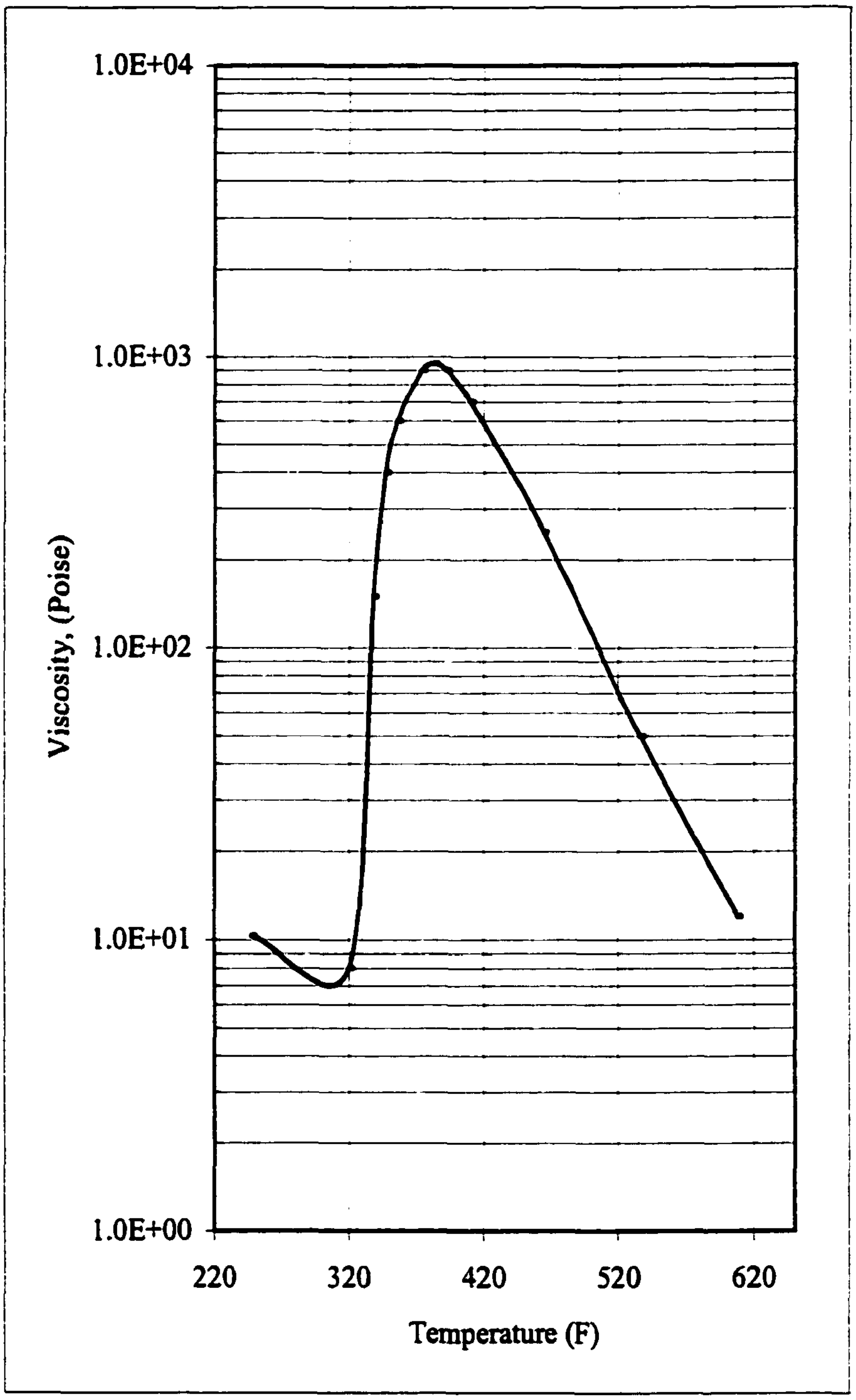


Figure 6: Viscosity of liquid sulfur, (Tobolsky, 1965)

Modified Sulfur

Elemental sulfur is known to exist in many forms but unfortunately the only form that is thermodynamically stable is the orthorhombic form S_α (Currell, 1976). Also, elemental sulfur has been proposed for a wide range of applications in the civil engineering field (Blight et al. 1977). In virtually all of these applications, it has been necessary to modify the sulfur with additives designed to prevent the embrittlement that occurs with pure elemental sulfur. Thus if sulfur is heated to 284 °C and then cooled to ambient temperature, monoclinic sulfur S_β is instantly formed. This process is followed by a reversion to orthorhombic sulfur S_α that is almost complete in 20 hours as shown in Figure 7.

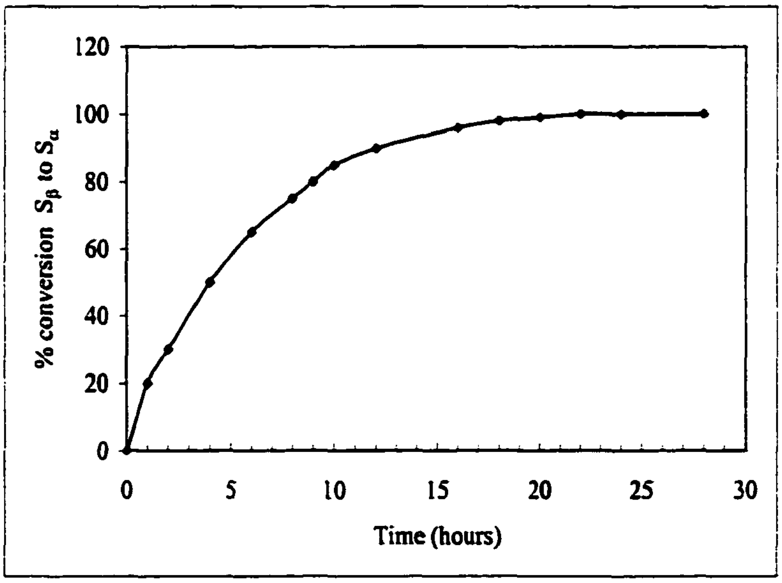


Figure 7: Reversion rate of monoclinic sulfur to orthorhombic sulfur at ambient temperature, (Blight et al. 1977)

To improve the behavior of solid sulfur, several additives have been proposed to modify elemental sulfur such that it becomes stable. Nearly all of these additives are one of several polymeric polysulfides or substances that react with elemental sulfur to form polymeric polysulfides. Modified sulfur is stable, i.e. there is no tendency for the sulfur to crystallize out. Some by-products of petroleum refining industry such as dicyclopentadiene, dipentene, styrene and methylcyclopentadiene dimer are suitable modifiers. The most stable formulation is sulfur modified with 6-7 percent by weight of dicyclopentadiene and 1 percent by weight of dipentene.

Production of modified sulfur involves heating and mixing the modifier and sulfur together. With rise of temperature, the reaction between elemental sulfur and modifiers begins and forms polymeric polysulfides. Reaction times range from 10 to 240 minutes, depending on temperature.

Unique properties of modified sulfur make it desirable for several construction purposes. Among its attributes are hardness, resistance to chemical attack, high compressive strength, low melting temperature, and low cost.

Sulfur Concrete

Since the late 1800's, sulfur cement and sulfur concrete have been investigated for use as a general construction material (Crick and Whitmore, 1998). The possible use of sulfur cement was based on its general characteristics of high compressive strength and exceptional resistance to chemical attack. However, it was not until mid 1970's that research was performed to modify sulfur to allow for the manufacture of sulfur concrete on a commercial scale basis.

As mentioned earlier sulfur concrete has excellent properties in terms of its resistance to corrosion. Table 5 shows the resistance of sulfur concrete to several corrosive chemicals at room temperature (Crick and Whitmore, 1998). The authors of the table set definite chemical concentrations for resistance of sulfur concrete to corrosion, however, setting a range for the concentration with time of application would have been more appropriate.

Sulfur concrete was used to make a floor in a refining plant (Funke and Mcbee, 1981). The floor was exposed to sulfuric acid byproducts. After 28 months of use, no corrosive deterioration occurred on the sulfur concrete floor sections. In comparison a control section was manufactured using regular Portland cement concrete and by comparison, this floor was severely deteriorated.

Sulfur concrete offers many benefits as an alternative construction material, such as rapid compressive strength development (Vroom, 1998), high compressive strength and resistance to freeze-thaw and resistance to abrasion when compared to Portland cement or polymer concrete. Sulfur can be stored in the open for indefinite periods without deterioration and the materials can be recycled by remelting with minor energy requirements. Table 6 shows a comparison between typical properties of sulfur concrete and Portland cement concrete, (Crick and Whitmore, 1998). In the original reference the authors set average values for typical parameters for Portland cement concrete, the table have been edited in order to provide a range for the properties instead of the average value.

Table 5: Chemical resistance to attack by corrosive chemicals at room temperature, (Crick and Whitmore, 1998).

Acids	Acid Concentration (percent)
Boric acid	100
Hydrochloric acid	32
Nitric acid	50
Phosphoric acid	85
Sulfuric acid	93
Chemicals	**Chemical Concentration (percent)**
Ammonium Sulfate	100
Calcium chloride	100
Copper sulfate	100
Ferric chloride	100
Magnesium chloride	100
Magnesium sulfate	100
Potassium chloride	100
Nickel chloride	100
Nickel sulfate	100
Sodium chloride	100
Sodium sulfate	100
Zinc chloride	100
Zinc sulfate	100

Table 6: Selected engineering properties of sulfur concrete in comparison with Portland cement concrete, edited (Crick and Whitmore, 1998)

Property	Typical results for Portland cement concrete	Typical results for sulfur Concrete
Compressive strength	4000-6000 psi	5000-9000 psi
Modulus of Rupture	500-900 psi	1850 psi
Tensile strength	190-390 psi	700-1000 psi
Modulus of elasticity	3-4 x 10^6 psi	4-6 x 10^6 psi
Density	150 pcf	150 pcf
Amount of binder	500 lb/yd^3	650 lb/yd^3
Setting time and curing	28 days	Not required

Due to sulfuric acid attack susceptibility, other alternatives have been used as alternatives for Portland cement concrete. These include Ductile Iron Pipe, Vitrified Clay pipe, Polyvinyl Chloride (PVC) pipe, HOBAS and T-lock lining for concrete pipes.

Vitrified clay pipe can not be used in all the applications due to the fact that the limited size limits using it and behavior in relation to reinforcement, ductile iron pipe is susceptible to acid attack with a higher cost. HOBAS is concrete pipe lined with fiberglass reinforced resin. The polyvinyl chloride (PVC) pipe is not available in large diameters because of limited crushing strength. Small diameters require special back fill provisions beside the fact that load distribution in the case of a flexible system is not fully understood. T-lock is PVC lining with r-shaped ribs, which are cast into reinforced concrete pipe. The lower size limit is because man entry is required to field weld the seams at joints.

Estimating cost of sewer construction is complex and involves many job specific variables (Pitt, 1995). However, it is possible to offer an estimate of the financial viability of the proposed process in comparison to alternative systems. Table 7 lists prices for different types of pipe delivered to Des Moines, IA

Table 7. Cost estimate of the variable sewer pipe alternatives available in the market, edited for polymer modified sulfur (Pitt, 1995)

Size, ID (inches)	Reinforced Concrete Pipe ($/ft)	HOBAS ($/ft)	PVC ($/ft)	Reinforced Concrete Pipe with T-lock ($/ft)	Sulfur Mortar Lined Concrete ($/ft)
30	26	60	42	N/A	29-31*
36	38	75	61	N/A	43-48*
42	51	95	N/A	94	56-62*
48	66	175	N/A	106	77-85*

* The first price for sulfur mortar lined pipe and the second price for DCP modified sulfur mortar lined pipe

Initial Experimentation with Sulfur Mortar for Lining Concrete Pipes

Initial experimentation was directed toward studying the engineering properties and feasibility of using sulfur mortar as a liner to resist acid attack and to define initial design parameters for the manufacturing process (Li, 1998).

Materials

A mixture of elemental sulfur and silica sand was used in this phase of the research. Type I Portland cement and silica sand from Ottawa, Illinois was used in preparing the concrete mortar specimens for abrasion tests for comparison purposes. Reagent grade sulfuric acid (H_2SO_4) diluted to 10% by volume was used to evaluate acid resistance. ASTM C 150-95a and ASTM C 778-92-a were used for cement and sand grading respectively.

Viscosity Test

A simple viscosity cup was developed to measure the viscosity of asphalt in the field. Results from this test field apparatus were in agreement with laboratory methods described by ASTM D 2170 and D 2171 (Potts, 1972). This test apparatus consists of two annular cups with a small tube that extends from the outside bottom into the inner cup. Both the interior cup and the annular space between the two cups are filled with molten sulfur. A 20-quart electrical heating pot was used to melt the sulfur to form the mortar.

Measuring the viscosity was performed by dipping the cup into the heating pot. The cup was allowed to heat for 10 minutes, then it was emptied 5 to 6 times to bring the cup to the temperature of the molten sulfur. Viscosity was determined by dipping the heated cup below the liquid surface, withdrawing the filled cup with a smooth vertical motion, and measuring the time it took the cup to empty. Testing results are presented in Figure 8. The trend line for sulfur mortar appeared different from that of pure sulfur. The curve shows that the efflux time can be classified into three phases within the range of test temperatures.

- Phase I: in this phase, the efflux time decreases with increase in temperature. In this phase, also, the state of modified sulfur transforms from solid into liquid with increase in temperature.
- Phase II: in this phase, the efflux time decreases slowly with increase in temperature. Using temperatures this phase showed an excellent bonding and workability properties for molding the material.

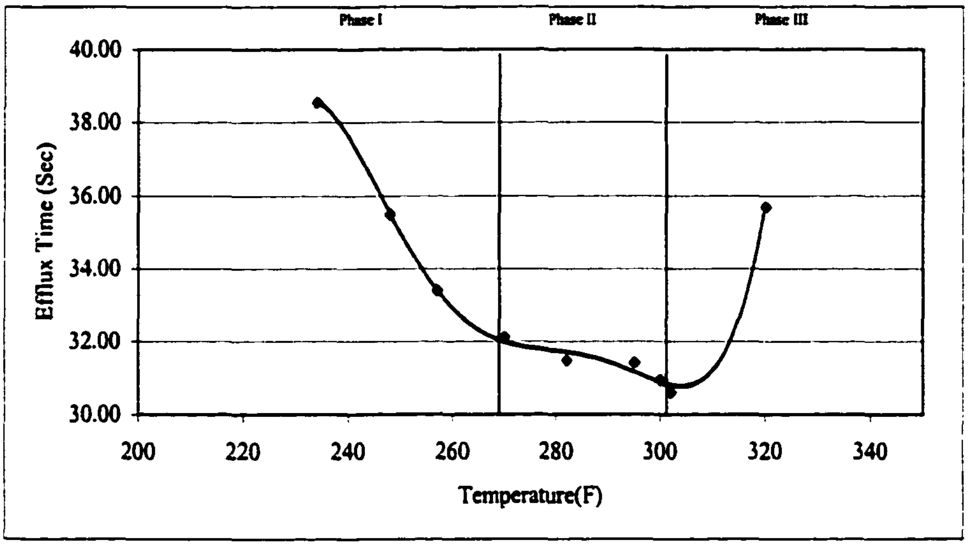


Figure 8: Relationship between efflux time of sulfur mortar and temperature (Li, 1998)

- Phase III: in this phase, the efflux time increases dramatically with increasing temperature. This is due to the fact that the composition of sulfur at this state of temperature is mainly in S_8 chains which is far longer than that of S_8 rings. This composition is responsible for increase in viscosity.

Bond Strength Test

Three factors were taken into consideration in the design of this study, moisture conditions of the substrates, temperature of molten sulfur mortar, and time of sample curing. Three moisture conditions of specimens for concrete substrates were selected. Saturated specimens were used to simulate concrete pipe coming directly from the autoclave. Concrete bricks were immersed in water at 77 °F, and weight changes were recorded several times until constant weight was obtained. Dry condition was studied; concrete bricks were dried in an oven at 230 to 239 °F for 24 hours. In ambient humidity condition, the saturated substrates were placed in open air for a period of time until the free water was in equilibrium with the (70±5)% relative humidity. This resulted in 3.70% moisture content.

In order to evaluate the effect of application temperatures of molten sulfur on the adhesive properties, bond strength was determined from specimens bonded with sulfur mortar at temperatures of 275 and 293 °F.

In order to determine the bond strength, test couplets were produced by pouring molten sulfur on the surface of one conditioned brick and immediately placing a second brick crosswise to the first brick. The resulting thickness of the mortar was in the order of 1/8". Actual measurements of thickness and failure mode are tabulated in Table 8. While testing bond strength, it was found that the bond strength was much greater than the strength of the concrete bricks. In an attempt to determine the true bond strength, the concrete bricks were reinforced by gluing two-1/16" thick steel plates to both sides of each brick with epoxy.

ASTM C 321-94 describes the standard procedure for determination of the bond strength between the mortar and the brick. The procedure involves placing a brick couplet in a special fixture that pushes the two bricks apart as illustrated by Figure 9. Six samples were tested for each condition and the average was estimated. A rate of loading of 0.20 in./min was used until failure was reached. Results of Testing are tabulated in Table 9.

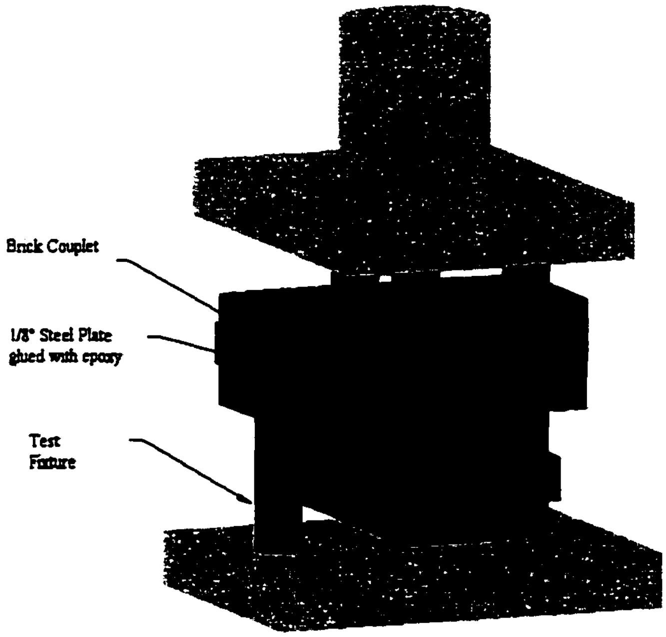


Figure 9: Schematic of the test fixture and the brick couplet

Time after bonding was considered as a significant variable; 90% of crystalline transformation is completed after 12 hours. Bond strength was tested after 1 hour, 24 hours and 28 days. ASTM C 190/C 190M-95 procedures were used to determine the compressive strength of the substrates. The average compressive strength of the six concrete substrates was 3445±245 psi. Typical Portland cement compressive strength is in the range of 4000 to 6000 psi, however, mortar bricks were used and these usually will posses lower than that of regular Portland cement concrete.

Three modes of failure were assessed while testing. Failure mode I is where the bond between the sulfur mortar and concrete is the weakest, failure mode II is where the adhesive capacity is greater than the shear strength of the concrete and failure mode III is where the bond strength exceeds the tensile capacity of the substrate. Analysis of the bond strength results indicated that in failure mode II, failure strength is always less than that of failure mode III. Also, mode I failure strength is always greater than failure mode II and mode III failure for specimens in ambient and oven-dry conditions or ambient humidity condition. Mode I failure bond strength in saturated condition is especially low. The following summarizes the effect of different conditions on bond strength testing:

- Effect of substrate conditioning method on bond strength:

Analysis of tables 8 and 9 indicates that samples that were oven-dry conditioned and were not reinforced had Mode II failure and all reinforced samples had Mode III failure. This is a good indication that the tensile strength of substrates is always greater than the shear strength of the substrates in the oven dry condition.

In addition, the experimental results show that the failure loads of both modes II and III are constant and independent of temperature and curing. In addition, at ambient humidity, no mode III failures occurred, suggesting that the tensile strength of the substrates is greater than that of the adhesion between concrete and sulfur. When the

Table 8: Thickness of sulfur mortar lining, conditions of curing, and failure mode (Li, 1998)

Sample No.	Thickness (in.)	Conditioning	Failure mode	Time of Curing	Temperature (°F)
1	0.24	Ambient humidity condition	I[i]	1 hour	273
2	0.28	Ambient humidity condition	I	1 hour	273
3	0.32	Ambient humidity condition	I	1 hour	295
4	0.28	Ambient humidity condition	I	1 hour	295
5	0.27	Ambient humidity condition	I	24 hour	295
6	0.29	Ambient humidity condition	I	24 hour	295
7	0.25	Ambient humidity condition	I	24 hour	273
8	0.28	Ambient humidity condition	I	24 hour	273
9	0.28	Ambient humidity condition	I	28 days	273
10	0.31	Ambient humidity condition	I	28 days	295
11	0.24	Ambient humidity condition	I	28 days	295
12	0.27	Ambient humidity condition	I	28 days	273
13	0.29	Oven dry condition	II[ii]	1 hour	273
14	0.26	Oven dry condition	II	24 hours	295
15	0.31	Oven dry condition	III[iii]	24 hours	273
16	0.26	Oven dry condition	II	28 days	295
17	0.30	Saturated condition	I	1 hour	273
18	0.25	Saturated condition	I	24 hours	273
19	0.29	Saturated condition	I	24 hours	295
20	0.26	Saturated condition	I	28 days	295

[i] The bond between the sulfur mortar and concrete is the weakest in the system.
[ii] Adhesive capacity is greater than the shear strength of the concrete.
[iii] The bond strength exceeded the tensile capacity of the substrate.

Table 9: Results and conditions for bond strength testing for sulfur mortar and concrete bricks (Li, 1998)

Time of Curing	Temperature of pouring (°F)	Conditioning	Steel Reinf.	Failure Mode	Mean Bond Strength (psi)
1 hour	273 °F	Oven Dry	No	II	46
			Yes	III	74
		Ambient Humidity	No	I	117
		Saturated	No	I	3
1 hour	295 °F	Oven Dry	No	II	48
			Yes	III	77
		Ambient Humidity	No	I	140
			No	II	122
			Yes	I	162
		Saturated	No	I	3
24 hours	273 °F	Oven Dry	No	II	50
			Yes	III	80
		Ambient Humidity	No	I	117
		Saturated	No	I	3
24 hours	295 °F	Oven Dry	No	II	47
			Yes	III	76
		Ambient Humidity	No	I	148
		Saturated	No	I	3
28 days	273 °F	Oven Dry	No	II	54
			No	I	106
28 days	295 °F	Oven Dry	No	II	53
			Yes	III	97
		Ambient Humidity	No	I	146

substrates are oven-dried, they lose their strength because of the heat. On the other hand, samples cured in ambient humidity fully developed their gel structure and strength and they developed mode I failure. Saturation of the substrates produced less strength in the case of mode I failure.

After comparing test results, it appears appropriate to use the ambient humidity conditioning for best practical results.

- Effect of time on bond strength:

Figure 10 shows the effect of time on bond strength for specimens cured at ambient temperature. When bonded at 293 °F, the difference between the 1 and 28 days was minimal.

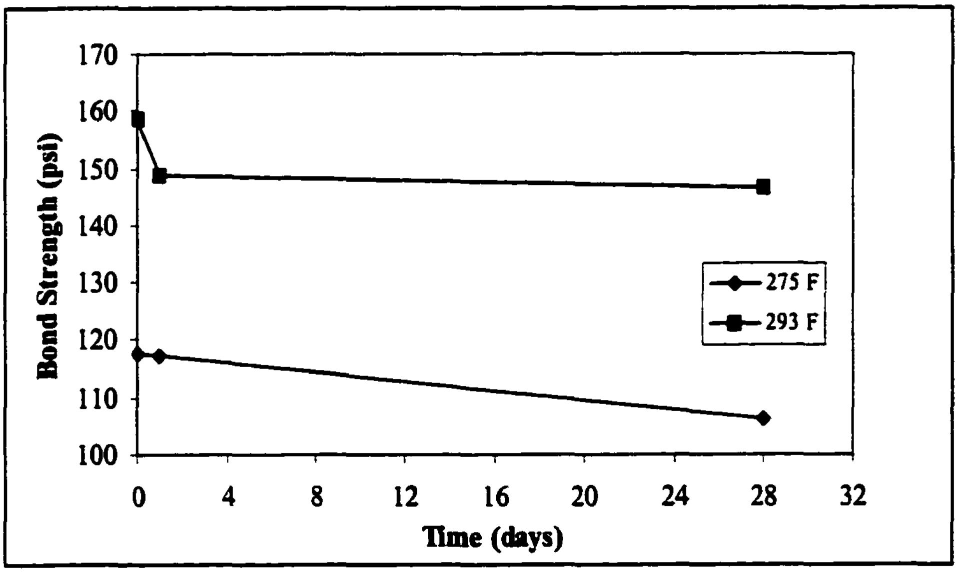


Figure 10: Development of bond strength with time for non-modified sulfur mortar (Li, 1998)

- Effect of temperature of molten sulfur on bond strength:

Figure 10 also illustrates the bond strength at 293 °F is always greater than that at 275 °F. Bond strength formed when material casting were developed at 293 °F is 28% greater than casting at 275 °F. Reduced strength at lower temperatures is the result of the mortar cooling before the second contact is complete.

Acid Penetration

Samples used for the study of the effectiveness of sulfur mortar as a barrier to acid were made by casting uniform mortar layers of 1/8", ¼", and ½" over conditioned concrete bricks. After solidification of the mortar, two rectangular plastic containers with an area of 7.56 in^2 were cut from the bottom and sealed to the solidified mortar surface with silicon compound. A 10% sulfuric acid solution was placed in the container with direct contact with the sulfur mortar surface. Initial pH of the solution was 0.25. Control samples without the sulfur mortar barrier were also prepared for comparison.

During acid penetration, visual observation and pH measurements were performed. No noticeable reaction occurred on the substrates covered with sulfur mortar. A noticeable amount of white solid products settled in the bottom and sidewalls of the acid containers of the control specimens. This white material is the product of the reaction between calcium carbonate and sulfuric acid. Values of pH were measured with time during testing. The pH remained constant for all samples with sulfur lining on them, while an increase in pH was noticed over time for the control specimens. Figure 11 gives the change in pH with time for both control and lined specimens. Test results showed that

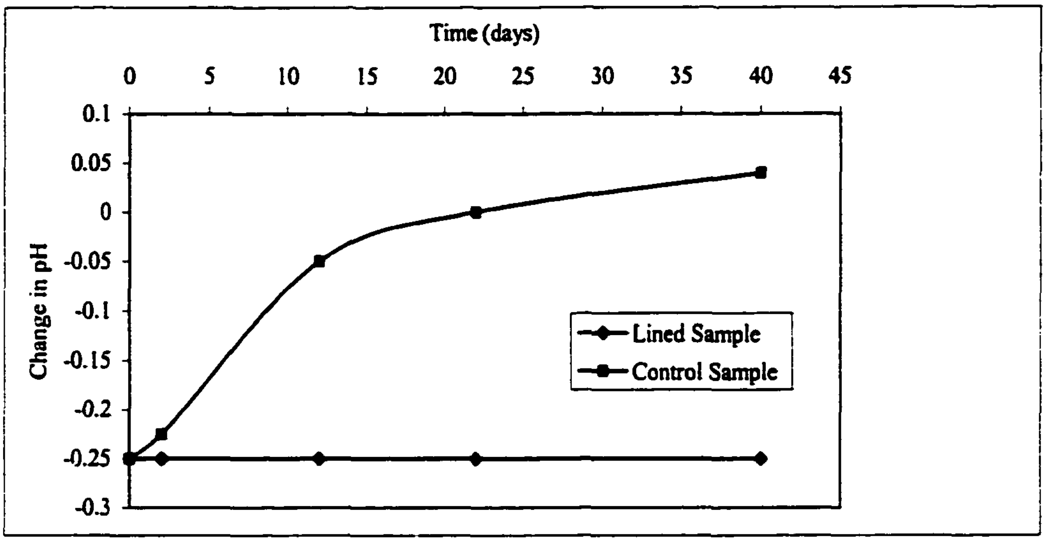


Figure 11: Effect of time on pH for concrete and sulfur lined bricks (Li, 1998)

a thickness of mortar of 1/8" is enough for protection of the substrates from sulfuric acid attack within test period.

As stated by the author (Li, 1998), the initial pH of the acid was -0.25. A negative value of pH is not possible. Typical values of pH range between 0 and 14 with a value of 7 set as neutral, pH greater than 7 is considered basic and pH less than 7 is considered acidic. The graph indicates that pH is increasing due to the reaction between sulfuric acid and calcium carbonate that will produce calcium sulfate that is a basic material which causes pH to increase.

Abrasion Resistance

ASTM C 1138-89 describes the standard test for abrasion resistance of concrete. This standard was used to facilitate a comparative evaluation of sulfur and Portland cement mortars. The apparatus used for this experiment consists of a motor and agitation paddle operation in water with an abrasive media. The specimen container was 4.88" in diameter and 7.75" in diameter. The abrasive media was steel ball bearings of the following numbers and diameters: 6 at 0.75", 14 at 0.5", and 20 at 0.25".

Test specimens were prepared of Sulfur mortar at 293 °F by pouring the molten mortar into cylindrical molds of 4.88" diameter and 0.81" thickness. Portland cement mortar specimens were prepared from a mixture of 2.75 parts sand to one part of cement using a water-cement ratio of 0.485. Two inches cube specimens were prepared of the Portland cement mortar. Those specimens were tested for compressive strength. The average compressive strength of the cubes was 1866, 2970, and 4412 psi at 3, 7, and 28 days respectively.

Testing was performed on cured samples. Curing was performed on sulfur specimens by immersion in water for a continuous 48 hours. Samples then were weighed in air and water in order to determine sample volumes, the specimens were then ground continuously for 12 hours, the abraded material was flushed, and the surface was wiped dry. The mass of the specimen was determined again in air and water. The test consisted of four 12- hour periods for a total of 48 hours.

Results of the abrasion test for both Portland cement mortar specimens and sulfur mortar are plotted in Figure 12. It can be seen that the abrasion rate during the first 12 hours is greater than those at other test periods for both materials. Also, it can be seen that the abrasion rate of sulfur mortar is less than that for Portland cement mortar.

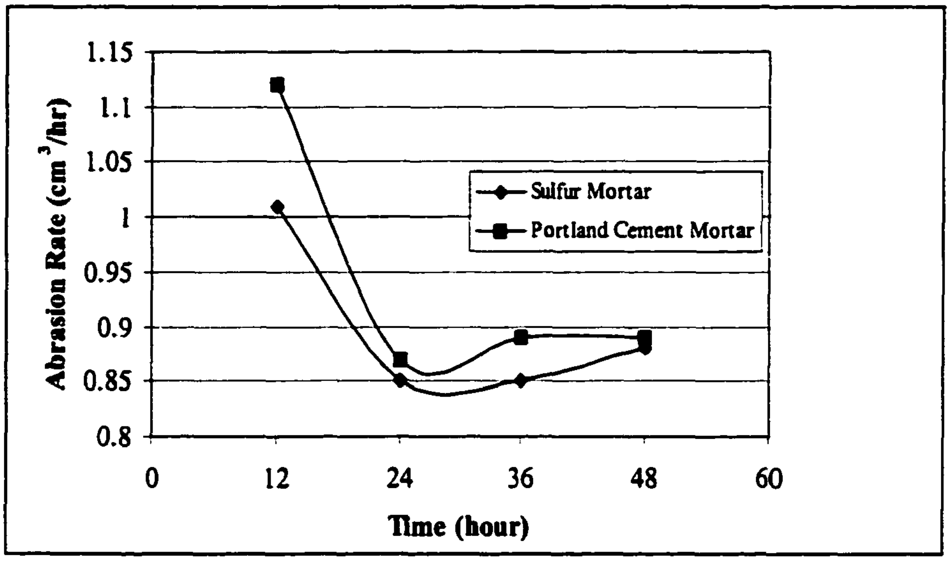


Figure 12: Comparison between abrasion rate of Sulfur and cement mortar (Li, 1998)

LABORATORY TESTING PROGRAM

Introduction

The objectives of the laboratory-testing program of the research were to evaluate the effects of polymer modification of sulfur mortar, its workability and ease of use when lining concrete sewer pipes. Initial experimentation with the sulfur mortar indicated that severe cracking occurred while lining concrete sewer pipe with the material. Additives studied include Dicyclopentadiene (3 and 5 % by weight) and fiberglass. This part presents the mechanical and engineering properties of the sulfur mortar and modified sulfur mortar. Engineering properties studied in this phase include compressive strength effect against aggregate content, bond strength, viscosity characteristics, resistance to sulfuric acid, and shrinkage characteristics.

Laboratory testing was performed to obtain the optimum additive content providing the best application properties in terms of compressive strength, bond strength, and the best application viscosity with least shrinkage of the material. Comparison between pure sulfur and polymer modified sulfur was also performed.

The compressive strength study of the laboratory-testing program was directed toward obtaining compressive strength equal or greater than that of concrete, i.e. 4000 to 6000 psi. Bond strength was also considered to assure proper bonding between the mortar and the concrete pipe. The viscosity of the sulfur mortar has a significant effect on the workability of the mortar. Applicable and practical workability range considered having a viscosity equivalent to emptying the cup within 50 to 100 seconds based on experience with the materials. This also assured that complete reaction between the polymer modifier and sulfur has occurred. Shrinkage of sulfur and sulfur mortars has a significant effect on the resulting cracking that the material will experience when it solidifies, reduction in shrinkage behavior was considered beneficial to the produced material.

Statistical analysis was performed on the laboratory testing results obtained in this study. Several independent variables were introduced in the laboratory-testing program in general, such as aggregate content, state of material in terms of modification, fiberglass content, time and temperature. Statistical analysis was used to evaluate each independent variable as being significant or not. Also, statistical analysis was used to evaluate the differences between the variable treatments in the same category and its effect on the resulting dependent variable, i.e. compressive strength, shrinkage and bond strength. The statistical analysis was performed using the Analysis of Variance (ANOVA) procedure with $\alpha=0.05$ to evaluate differences between the means of the dependent variables, the difference between the variable treatments results was evaluated using the Tukey's and pairwise t-tests. Results of the statistical analyses are shown in Appendix B.

Materials

A mixture of elemental sulfur, fine silica sand extracted from silica crusher fines, and Dicyclopentadiene sulfur modifier was used. Reagent grade sulfuric acid (H_2SO_4) diluted to 10% by volume was used to evaluate acid resistance. Modified Sulfur was prepared by heating the sulfur using a 20-quart heating pot to the reaction temperature, 280 °F, the modifier was then added to the sulfur. The polymer and sulfur were left for two hours for the reaction to be completed, and then the aggregates were added to the mixture after 1 ¾ hours of reaction time.

Compressive strength characterization

Compressive strength testing was performed in accordance with ASTM C 579-91. Testing was performed on 2x2x2" cube samples of sulfur, and 3% and 5% DCP modified sulfur while varying the aggregate content from 0 to 50% of the weight of the sulfur. Three (3) samples were tested for each condition. Applied fiberglass reinforcement was 1% of the total weight of the sulfur mortar or the modified sulfur mortar. The samples were cured by leaving them at ambient temperature and humidity. Samples were tested for compressive strength after 1 hour, 24 hours, 3 days, 7 days and 14 days.

In the process of the preparation of the samples, an upper reservoir of the material was provided to compensate for the shrinkage of the material. Three samples were prepared and tested for each time interval, and then the mean and standard deviation of the test results were plotted with time. Figures 13 through 18 show the results of the compressive strength tests for similar materials while varying the aggregate content.

Analyses of test results obtained for pure sulfur plus aggregate as illustrated in Figure 13 show that pure sulfur mortar will gain strength up to 3 days. After 3 days there is a decrease in the compressive strength with time. This is mainly due to the transformation to the crystalline state. On the other hand, reinforcement of pure sulfur mortar with fiberglass resulted in lesser reduction in compressive strength with time as can be seen in Figure 13.

Modification of sulfur using 3% and 5% DCP improved the compressive strength significantly and no significant decrease in compressive strength with time was noticed. Increasing the aggregate content increased the compressive strength of the material in general for all cases studied, but this increase should be balanced with the viscosity and level of workability needed.

Reinforcement of the sulfur and modified sulfur mortar with fiberglass will increase the compressive strength of materials used; however, this increase is not high with larger aggregate contents. Greater increase in compressive strength was noticed at lower aggregate contents as can be seen by comparing Figures 15 and 16 with Figures 18 and 19, respectively. Less reduction in compressive strength with time was noticed when using fiberglass reinforcement with the various mixtures as indicated by the lesser declining slope.

Statistical analyses of differences for the compressive strength testing are shown in Tables 10 through 14. Statistical analyses of compressive strength testing results show that all independent variables (aggregate content, fiberglass content, time of curing, and material state of modification) involved are significant as shown in Table 10.

The differences between the variable treatments within the one independent variable were significant for all the treatments except the time of curing as can be seen in Table 11. It was noted that there is no significant difference between results obtained after 72, 168 and 336 hours of preparation, however, there is a significant difference between the 1 and 24 hour and the 336 and 168 hours.

This also suggests that reduction in compressive strength that occurs with time have been eliminated. The overall time factor remains significant.

Table 10: Statistical analysis of the effect of the various independent variables involved in compressive strength testing

R-Square		Coefficient of Variation	Compressive Strength Mean
1.0		6.8	4763.0
Source	DOF	F Value	Pr>F
Replicate (Compressive Strength)	2	2.9	0.0560
Fiberglass Content	1	1140.9	<0.0001
Aggregate Content	5	2429.5	<0.0001
Material (Sulfur, 3% and 5% Modified)	2	1193.2	<0.0001
Time	4	449.5	<0.0001

Compressive strength of sulfur mortars with 30% or more aggregate resulted in compressive strength similar to or greater than that of Portland cement concrete. Compressive strength of 3% DCP modified sulfur mortars with 10% or more aggregate resulted in compressive strength similar to greater than that of Portland cement concrete. 5% DCP did not result in improvement in the compressive strength of the material. As mentioned earlier, the research was directed to obtaining compressive strength similar or greater than that of Portland cement concrete, modification with 3% DCP with 10 % or more aggregate resulted in less loss of compressive strength and at the same time compressive strength greater than that of concrete. 3% DCP modified sulfur mortar plus 50% aggregate appeared to posses excellent compressive strength properties and at the same time gave good workability, for the previous reasons, the later mixture was selected to be used as a base liner.

Table 11: Summary of statistical analysis of the differences in the compressive strength due to the time factor if significant or not using the pairwise t-test

	1	24	72	168	336
1	---	Yes	Yes	Yes	Yes
24	Yes	---	Yes	Yes	Yes
72	Yes	Yes	---	No	No
168	Yes	Yes	No	---	No
336	Yes	Yes	No	No	---

Table 12: Summary of statistical analysis of the differences in the compressive strength due to the fiberglass content factor if significant or not using the pairwise t-test

	1	0
1	---	Yes
0	Yes	---

Table 13: Summary of statistical analysis of the differences in the compressive strength due to the aggregate content factor if significant or not using the pairwise t-test

	0	10	20	30	40	50
0	---	Yes	Yes	Yes	Yes	Yes
10	Yes	---	Yes	Yes	Yes	Yes
20	Yes	Yes	---	Yes	Yes	Yes
30	Yes	Yes	Yes	---	Yes	Yes
40	Yes	Yes	Yes	Yes	---	Yes
50	Yes	Yes	Yes	Yes	Yes	---

Table 14: Summary of statistical analysis of the differences in the compressive strength due to the state of modification factor if significant or not using the pairwise t-test

	Sulfur Mortar	Sulfur + 3% DCP	Sulfur + 5% DCP
Sulfur Mortar	---	Yes	Yes
Sulfur + 3% DCP	Yes	---	Yes
Sulfur + 5% DCP	Yes	Yes	---

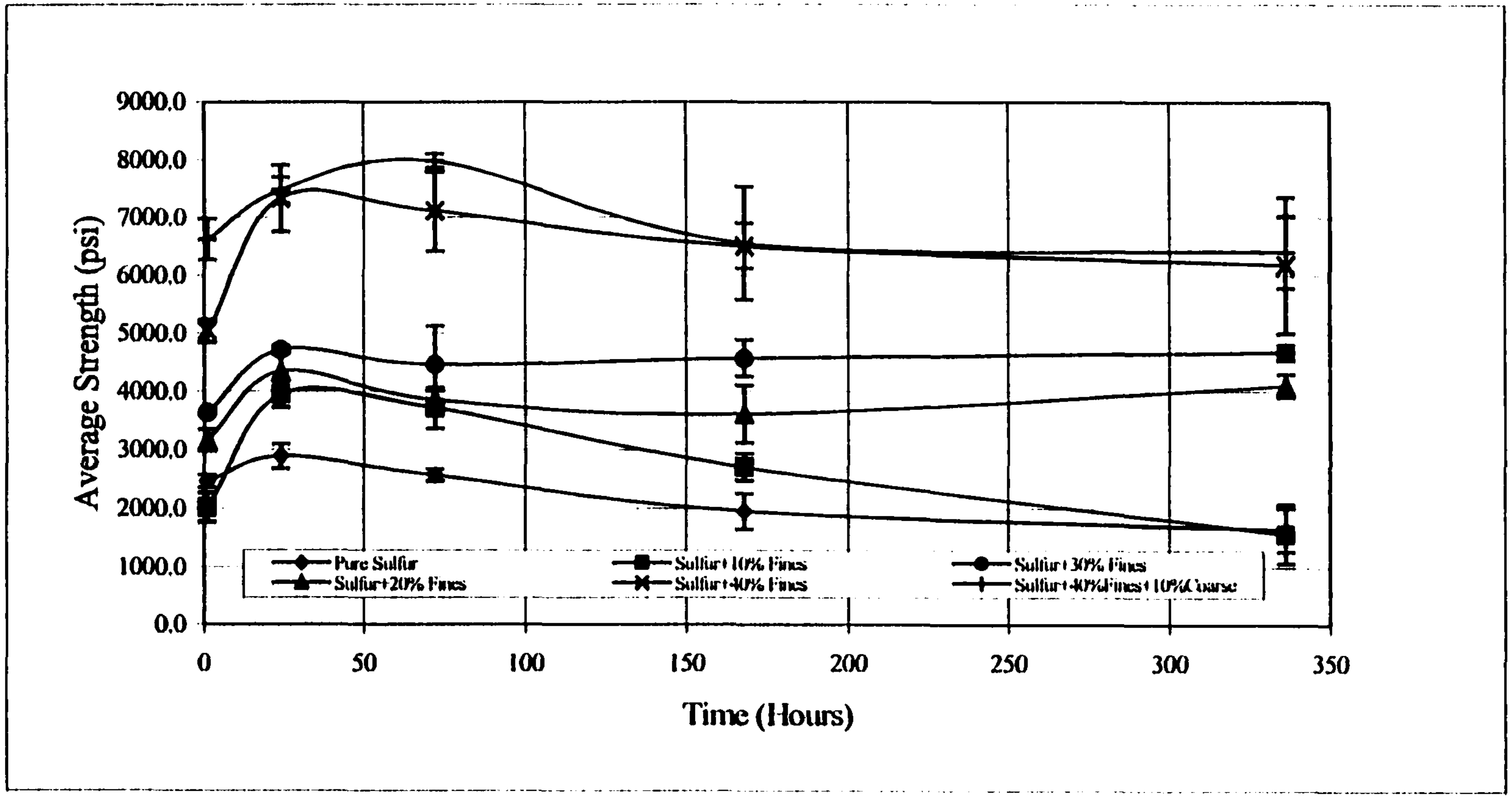


Figure 13: Compressive strength versus time for different mixtures of pure sulfur

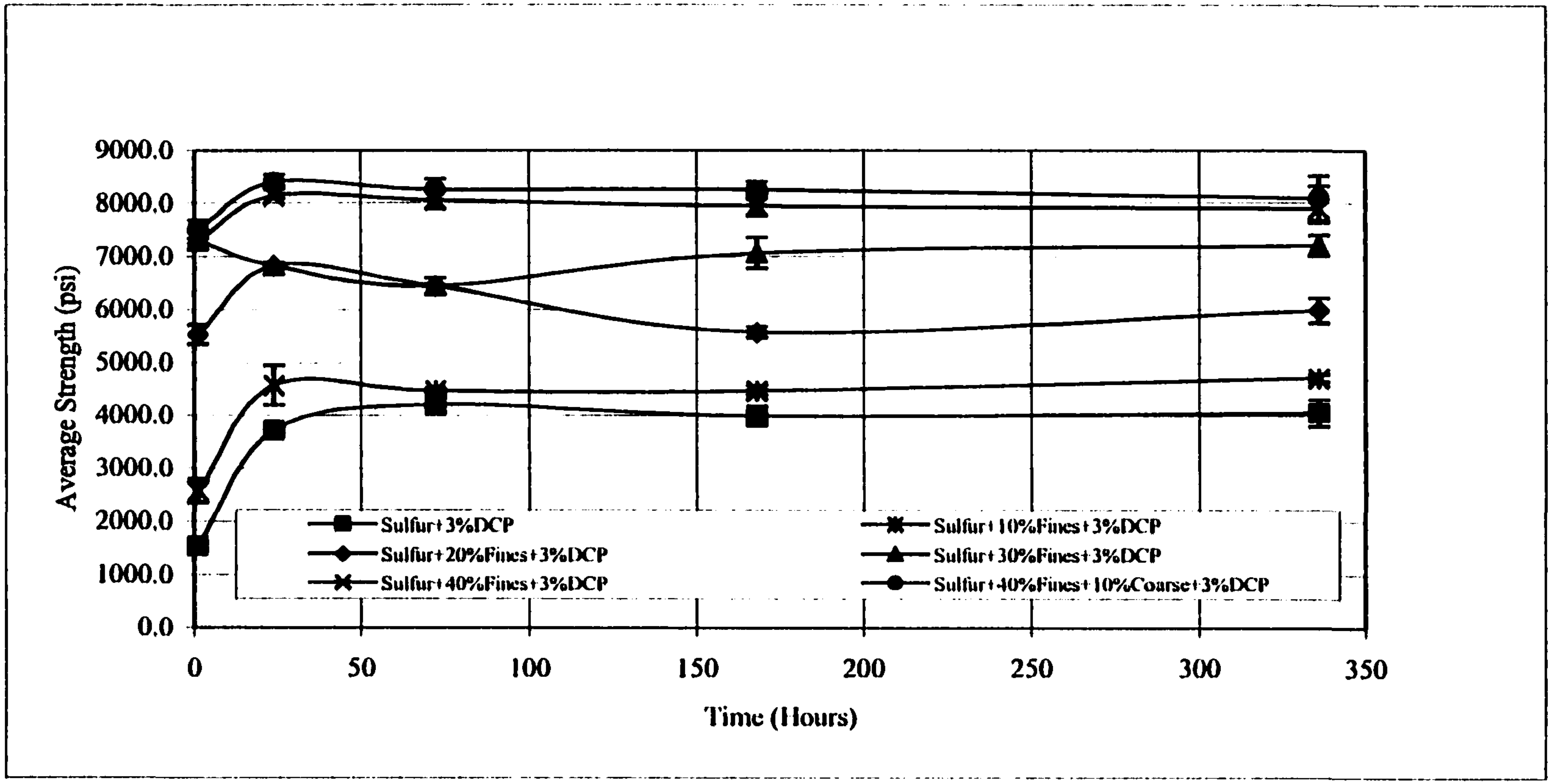


Figure 14: Compressive strength versus time for different mixtures of 3% DCP modified sulfur

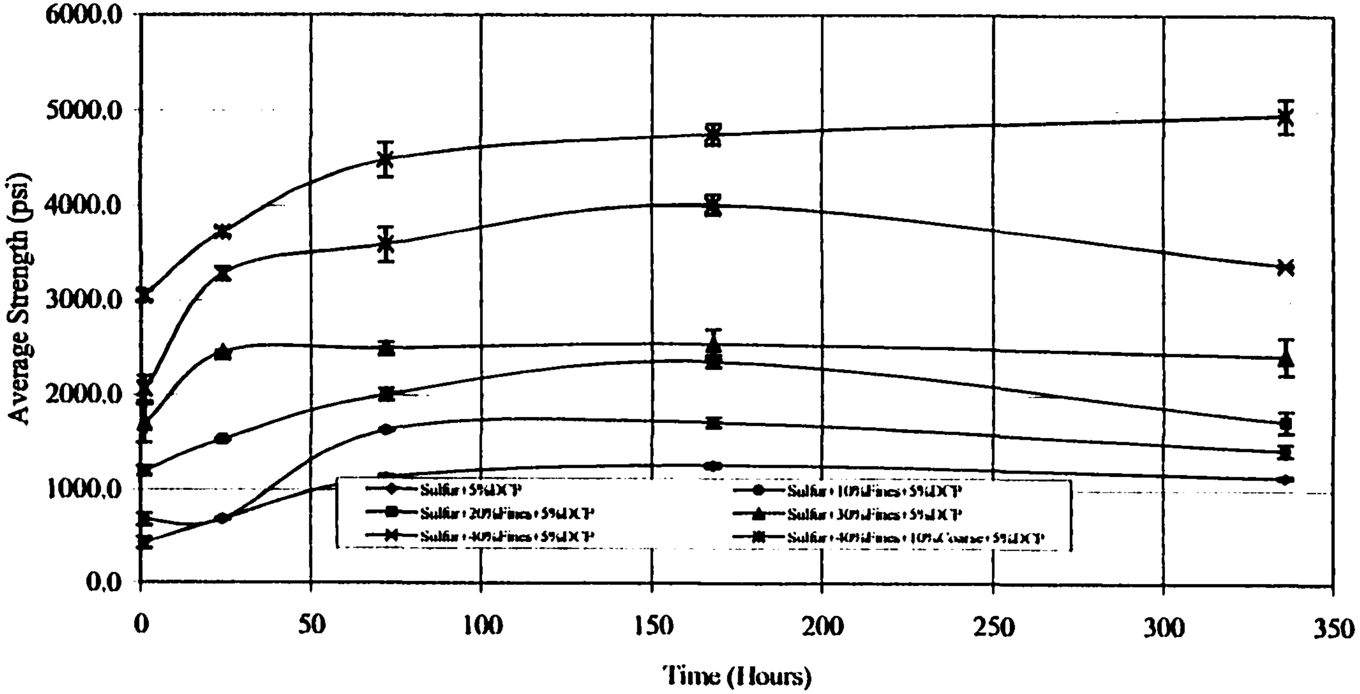


Figure 15: Compressive strength versus time for different mixtures of 5% DCP modified sulfur

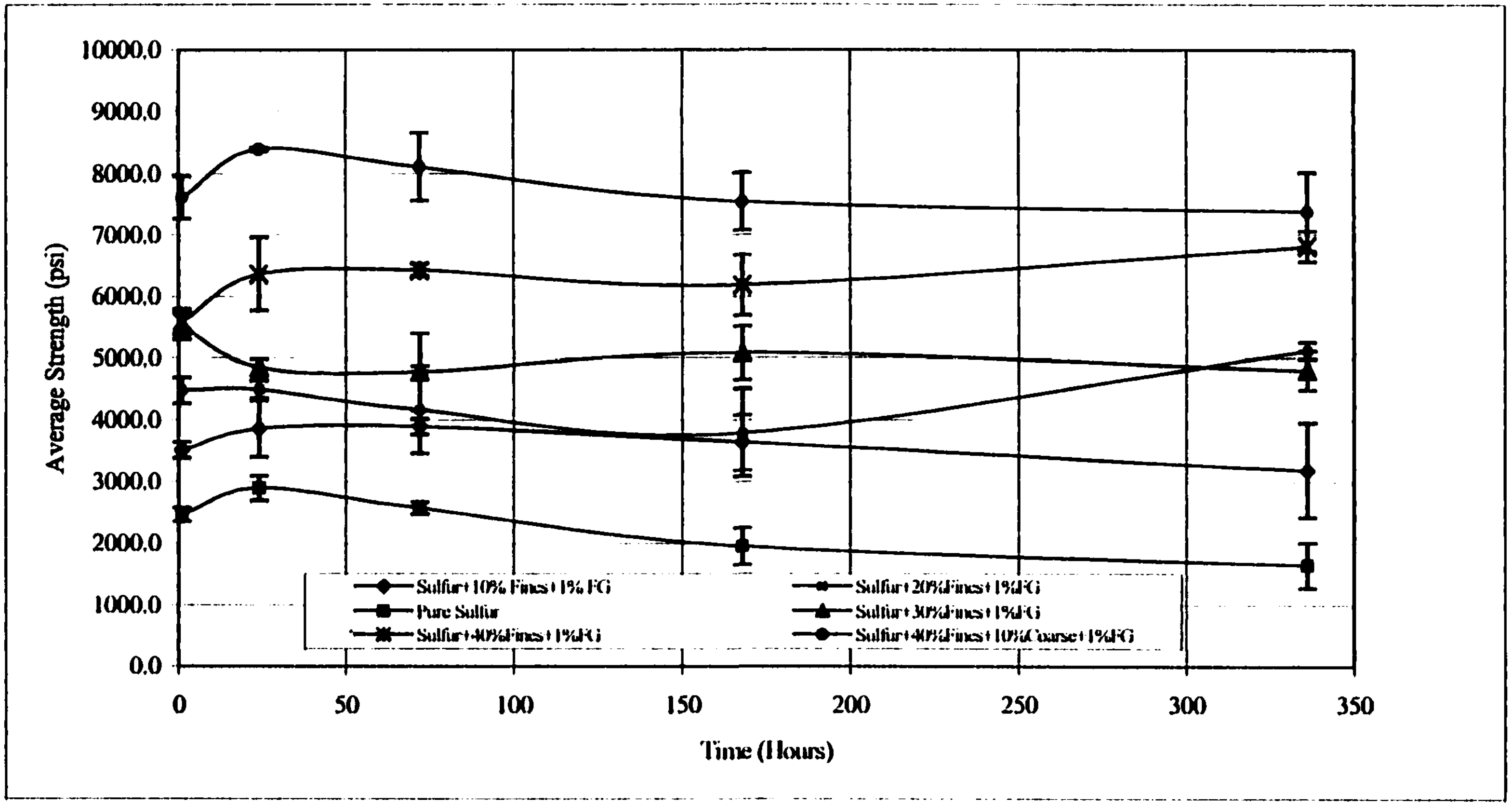


Figure 16: Compressive strength versus time for different mixtures of elemental sulfur mortar with fiberglass reinforcement

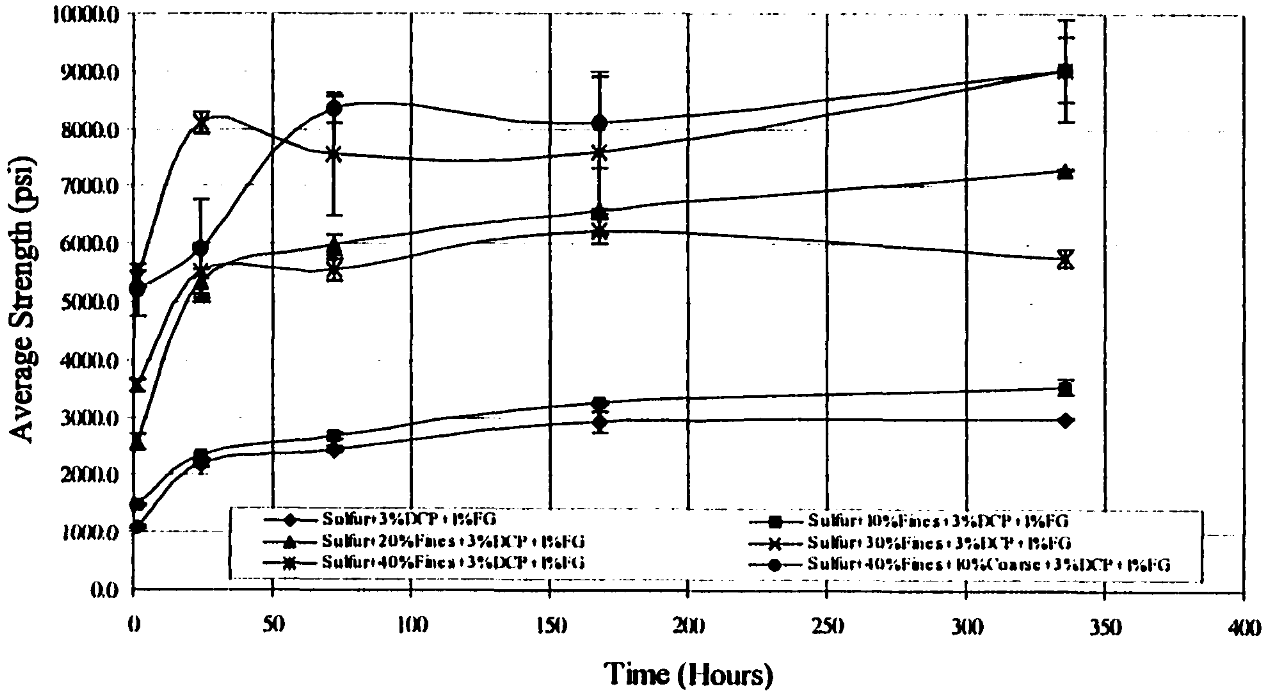


Figure 17: Compressive strength versus time for different mixtures of 3% modified sulfur mortar with fiberglass reinforcement

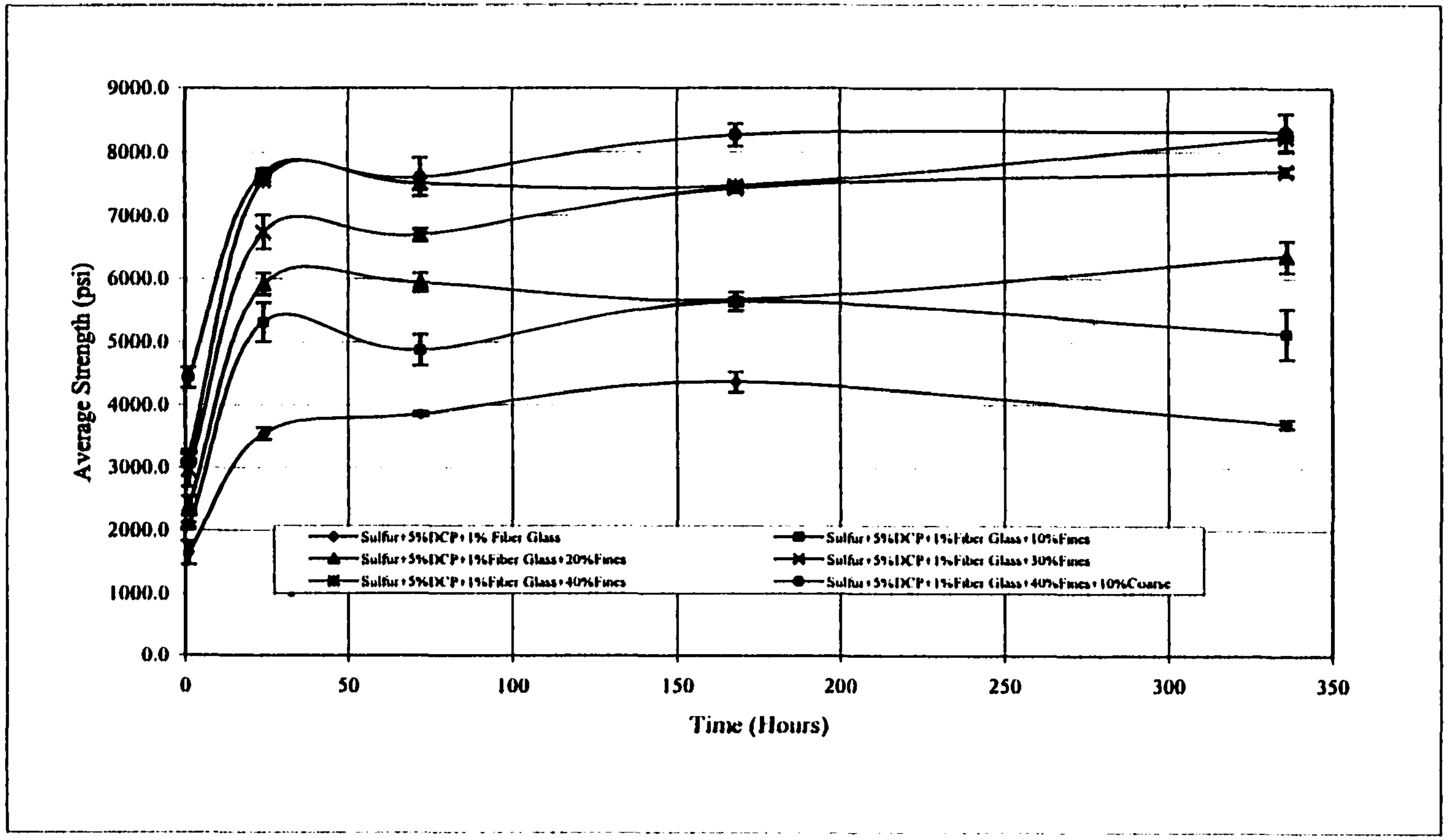


Figure 18: Compressive strength versus time for different mixtures of 5% modified sulfur mortar with fiberglass reinforcement

Viscosity Test

The same procedures and apparatus used in to perform viscosity testing described in the literature review (Li, 1998) were used for testing of materials in this phase. The efflux time was measured as indication of the viscosity if materials. This test was performed to aid in evaluating the effect of mixing time of the polymer. Testing results are presented in Figure 19 for reaction time versus the viscosity for 3 and 5% DCP used in the modification process.

It was observed that the viscosity increases with increasing reaction time. The viscosity was not measured after reaction time of 4 and 6 hours for 3 and 5% DCP, respectively since the viscosity became extremely high such that the method used was no longer applicable. In addition, it can be seen that using 3% modification gave an extended period to work with the material and a reasonable viscosity to obtain a workable mix.

Another test was performed to correlate the viscosity with the aggregate content. No realistic correlation was obtained for this test. The factors affecting the test included the measured time of flow and the temperature variation of the heating mandrel. In addition, looking at the overall average for all tests for the time of flow that was approximately 21.25 seconds and the standard deviation of the time of flow that ranged between tests of 0.5 to 2 seconds. As a result, there will be a significant effect for the error on the results of this experiment. The results indicate that this test or procedure is not suitable for testing the viscosity versus the aggregate content, and other methods may be more suitable. The results are illustrated in Figure 20.

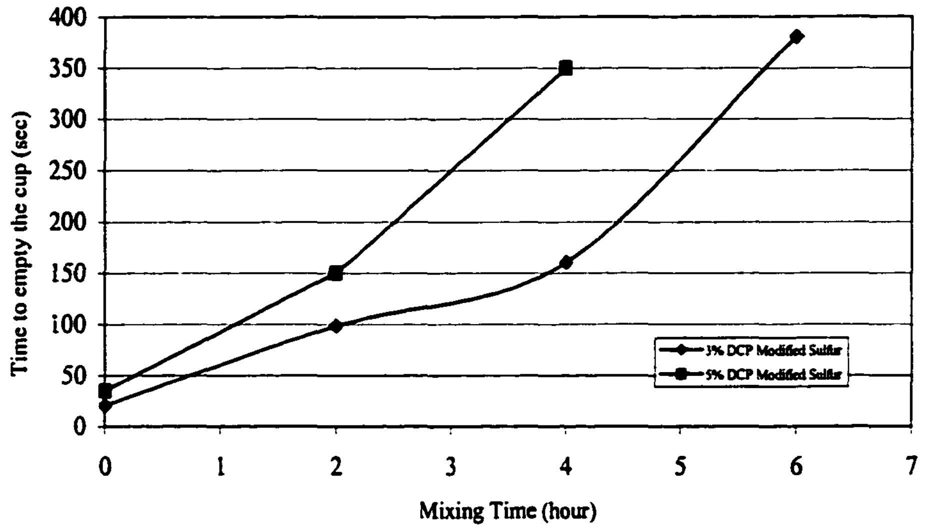


Figure 19: Effect of reaction time on viscosity of the two polymer concentrations used

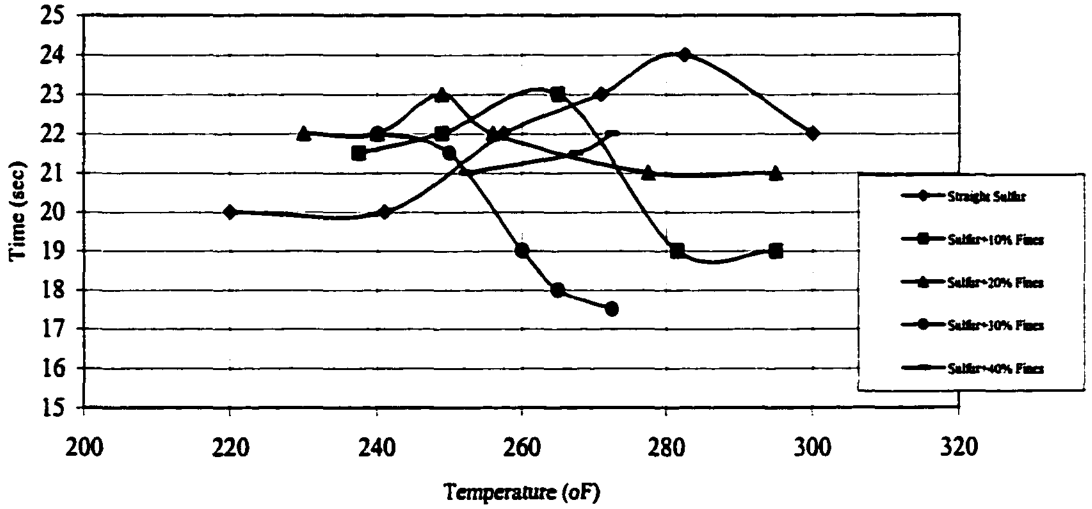


Figure 20: Effect of aggregate content on viscosity

Experience with the material indicated that a workable (pourable) mix would be obtained when the efflux time of the material is less than 100 seconds and preferably less than 75 seconds. Mixtures of sulfur mortar and 3% DCP modified sulfur mortars conformed to the previous time criterion and they provided excellent workability behavior.

Bond Strength Test

In the initial experimentation with sulfur in the literature review section, it was illustrated that best bond between the sulfur and concrete will be obtained if pouring occurred at ambient humidity condition (Li, 1998). The temperature of molten modified sulfur mortar was the only variable taken into consideration in the design of this study in order to evaluate the effect of application temperatures of molten sulfur on adhesive properties. Also, in this phase, testing was directed toward obtaining mode I failure (In an attempt to determine the true bond strength, the concrete bricks were reinforced by gluing 1/16" thick steel plates to each side of the two bricks with epoxy)., i.e. when the bond is less than the tensile capacity of the substrate.

Bond strength was determined from specimens bonded with modified and unmodified sulfur mortar at temperatures of 275 and 290 °F. For this purpose, test couplets were produced by pouring a ladle of molten sulfur on the surface of one conditioned brick and immediately placing a second brick crosswise to the first brick. The resulting thickness of the mortar was in the order of 1/8". Three bond strength samples were tested for each condition. Samples were tested 24 hours after preparation.

ASTM C 321-94 describes the standard procedure used to determine the bond strength. The procedure involves placing a brick couplet in a special fixture that pushes the two bricks apart. A rate of loading of 0.20 in./ min was used until failure load was reached.

Test results are tabulated in Tables 15 and 16. Statistical analyses of the differences between means of bond strength were performed to check if significant differences exist between the variable treatments. Results of the significance of the independent variables involved are presented in Table 17, the results of the significance testing indicate that fiberglass content and pouring temperature are not significant using 95% confidence interval. All the remaining independent variables were shown to be significant. The differences studies for the effect within the single independent variable are presented in Tables 18 through 21.

The test results indicate that bond strength at a pouring temperature of 290 °F is slightly greater than that at 275 °F for pure sulfur mortar. On the other hand, lower pouring temperature resulted in slightly higher bond strength in the case of modified sulfur mortars. Statistical analyses of bond strength results show that there are no significant difference as a result of changing the pouring temperature, and this is shown by the slight increase in the bond strength.

Also, it can be seen that 3% DCP modification gave greater bond strength than 5% DCP modification and statistical analyses proved that there are differences due to varying the polymer content.

Table 15: Bond strength results for 275 °F pouring temperature

Material	Average Bond Strength (psi)	Failure Mode
Sulfur	76	I
Sulfur +50% Aggregate	136	I
Sulfur +50% Aggregate + 1 % Fiber Glass	128	I
Sulfur +3% DCP	152	I
Sulfur +3% DCP + 50% Aggregate	170	I
Sulfur +3% DCP + 50% Aggregate + 1% Fiber Glass	127	I
Sulfur +5% DCP	124	I
Sulfur +5% DCP + 50% Aggregate	162	I
Sulfur +5% DCP + 50% Aggregate + 1% Fiber Glass	134	I

Table 16: Bond strength results for 290 °F pouring temperature

Material	Average Bond Strength (psi)	Failure Mode
Sulfur	120	I
Sulfur +50% Aggregate	161	I
Sulfur +50% Aggregate + 1% Fiber Glass	125	I
Sulfur +3% DCP	128	I
Sulfur +3% DCP + 50% Aggregate	160	I
Sulfur +3% DCP + 50% Aggregate + 1% Fiber Glass	162	I
Sulfur +5% DCP	126	I
Sulfur +5% DCP + 50% Aggregate	144	I
Sulfur +5% DCP + 50% Aggregate + 1% Fiber Glass	130	I

The presence of fiberglass in pure sulfur mortar or in modified sulfur did not result in a consistent effect, in one case it resulted in increasing bond strength and in another it resulted in decreasing bond strength. Statistical analyses proved that the presence of fiberglass is insignificant to bond strength.

The presence of aggregate in the pure sulfur mortar or in modified sulfur mortar tends to increase the bond strength in all cases and this is proven by statistics by the significant difference between the different treatments used in the experiment.

Table 17: Statistical analysis of the effect of the various independent variables involved in bond strength testing

R-Square	Coefficient of Variation	Bond Strength Mean (psi)
0.85	8.5	137.4

Source	DOF	F Value	Pr>F
Replicate (Bond Strength)	2	0.8	0.4396
Fiberglass Content	1	1.4	0.2541
Aggregate Content	1	50.6	<0.0001
Material (Sulfur, 3% and 5% Modified)	2	21.4	<0.0001
Temperature	1	2.6	0.1168

Table 18: Summary of statistical analysis of the differences in the bond strength due to the fiberglass content factor if significant or not using the pairwise t-test

	1	0
1	---	No
0	No	---

Table 19: Summary of statistical analysis of the differences in the bond strength due to the temperature factor if significant or not using the pairwise t-test

	270	290
270	---	No
290	No	---

Table 20: Summary of statistical analysis of the differences in the bond strength due to the aggregate content factor if significant or not using the pairwise t-test

	0	50
0	---	Yes
50	Yes	---

Table 21: Summary of statistical analysis of the differences in the bond strength due to the state of modification factor if significant or not using the pairwise t-test

	Sulfur Mortar	Sulfur + 3% DCP	Sulfur + 5% DCP
Sulfur Mortar	---	Yes	Yes
Sulfur + 3% DCP	Yes	---	Yes
Sulfur + 5% DCP	Yes	Yes	---

Shrinkage Characteristics

Shrinkage of sulfur is one of the most important factors involved in using sulfur concrete or sulfur mortar as a civil engineering material because shrinkage results in cracking. Therefore, it was an essential step to estimate and evaluate the shrinkage characteristics of the material. Studying the shrinkage characteristics was performed to assist in designing the molds that will be used to modify the existing concrete pipe and to compensate for the shrinkage effect of the material.

Testing was performed in accordance with ASTM C 531-85. In this method, a 1x1x10-inch bar is prepared using a specially designed mold. Two contact points (precision screws) are installed at each end of the bar, the bar then is allowed to cure, and the change in length with time is then measured using a comparator. Two samples were prepared for each mixture. Testing was performed with time and average values of the results were estimated. The comparator allows measurement of shrinkage using an accuracy of 1/10000 inch.

This standard procedure does not compensate for the initial shrinkage that occurs during the initial solidification of the sulfur mortar. A feeler gauge was used to evaluate the initial shrinkage that occurs immediately after the solidification of the sulfur mortar bar while the bar is still in the mold. The bar then was released from the mold and the comparator was used to evaluate the shrinkage thereafter. The results of both procedures were added together in order to evaluate the total shrinkage. Results of shrinkage testing are presented in Figures 21, 22 and 23.

Analyses of test results show that the pure sulfur mortar will experience more shrinkage than modified sulfur mortar. Also, it can be seen that 3% DCP modified sulfur mortar will result in more shrinkage than that of the case using 5% modification. The presence of aggregate in the mixture will reduce the amount of shrinkage in all cases. Fiberglass reinforcement resulted in less shrinkage in all cases included in this study.

Statistical analyses of the differences between means of shrinkage were performed to check if significant differences exist between the variable treatments. Results of the significance of the independent variables involved are presented in Table 22. Results shown in Table 22 indicate all the independent variables are significant. The difference studies for the effect within the single independent variable are presented in Tables 23 through 26.

Analyses of variance of testing results indicated that all parameters involved in the experiment such as aggregate content, time, modification and fiberglass content are significant and they have a significant effect on shrinkage results as can be seen from Table 22. However, the time factor becomes insignificant after 72 hours, and this leads to the conclusion that shrinkage becomes negligible after 72 hours after preparation of the samples as can be seen in Table 26.

As mentioned earlier, reduction in shrinkage will result in less cracking, and this is beneficial to the overall lining process. Quantitative analyses of the results indicate that the presence of 50% aggregate in general will result in approximately 40% reduction in the percent change in length. Polymer modification of the material will result in approximately 10 to 20% reduction in the percent change in length. As mentioned earlier, any reduction in shrinkage of sulfur would be beneficial to this research, it can be seen that the modification of sulfur resulted in less shrinkage and visual examination of samples prepared indicated no cracking of the polymer modified sulfur mortars.

Table 22: Statistical analysis of the effect of the various independent variables involved in shrinkage testing

R-Square		Coefficient of Variation	Bond Strength Mean
1.0		4.3	0.2
Source	DOF	F Value	Pr>F
Replicate (Shrinkage)	1	20.0	<0.0001
Fiberglass Content	1	4788.0	<0.0001
Aggregate Content	1	7978.9	<0.0001
Material (Sulfur, 3% and 5% Modified)	2	5743.7	<0.0001
Time	18	532.0	<0.0001

Table 23: Summary of statistical analysis of the differences in the shrinkage due to the fiberglass content factor if significant or not using the pairwise t-test

	1	0
1	---	Yes
0	Yes	---

Table 24: Summary of statistical analysis of the differences in the shrinkage due to the state of modification factor if significant or not using the pairwise t-test

	Sulfur Mortar	Sulfur + 3% DCP	Sulfur + 5% DCP
Sulfur Mortar	---	Yes	Yes
Sulfur + 3% DCP	Yes	---	Yes
Sulfur + 5% DCP	Yes	Yes	---

Table 25: Summary of statistical analysis of the differences in the shrinkage due to the aggregate content factor if significant or not using the pairwise t-test

	0	50
0	---	Yes
50	Yes	---

Table 26: Summary of statistical analysis of the differences in the shrinkage due to the time factor if significant or not using the pairwise t-test

	0.15	0.5	0.67	0.83	1	1.5	2	2.5	3	4	8	24	48	72	96	120	144
0.15	---	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes
0.5	Yes	---	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes
0.67	Yes	Yes	---	No	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes
0.83	Yes	Yes	No	---	No	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes
1	Yes	Yes	Yes	No	---	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes
1.5	Yes	Yes	Yes	Yes	Yes	---	No	No	No	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes
2	Yes	Yes	Yes	Yes	Yes	No	---	No	No	No	Yes	Yes	Yes	Yes	Yes	Yes	Yes
2.5	Yes	Yes	Yes	Yes	Yes	No	No	---	No	No	Yes	Yes	Yes	Yes	Yes	Yes	Yes
3	Yes	Yes	Yes	Yes	Yes	No	No	No	---	No	Yes	Yes	Yes	Yes	Yes	Yes	Yes
4	Yes	Yes	Yes	Yes	Yes	Yes	No	No	No	---	Yes	Yes	Yes	Yes	Yes	Yes	Yes
8	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	---	Yes	Yes	Yes	Yes	Yes	Yes
24	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	---	No	Yes	Yes	Yes	Yes
48	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	No	---	No	No	No	No
72	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	No	---	No	No	No
96	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	No	No	---	No	No
120	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	No	No	No	---	No
144	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	No	No	No	No	---

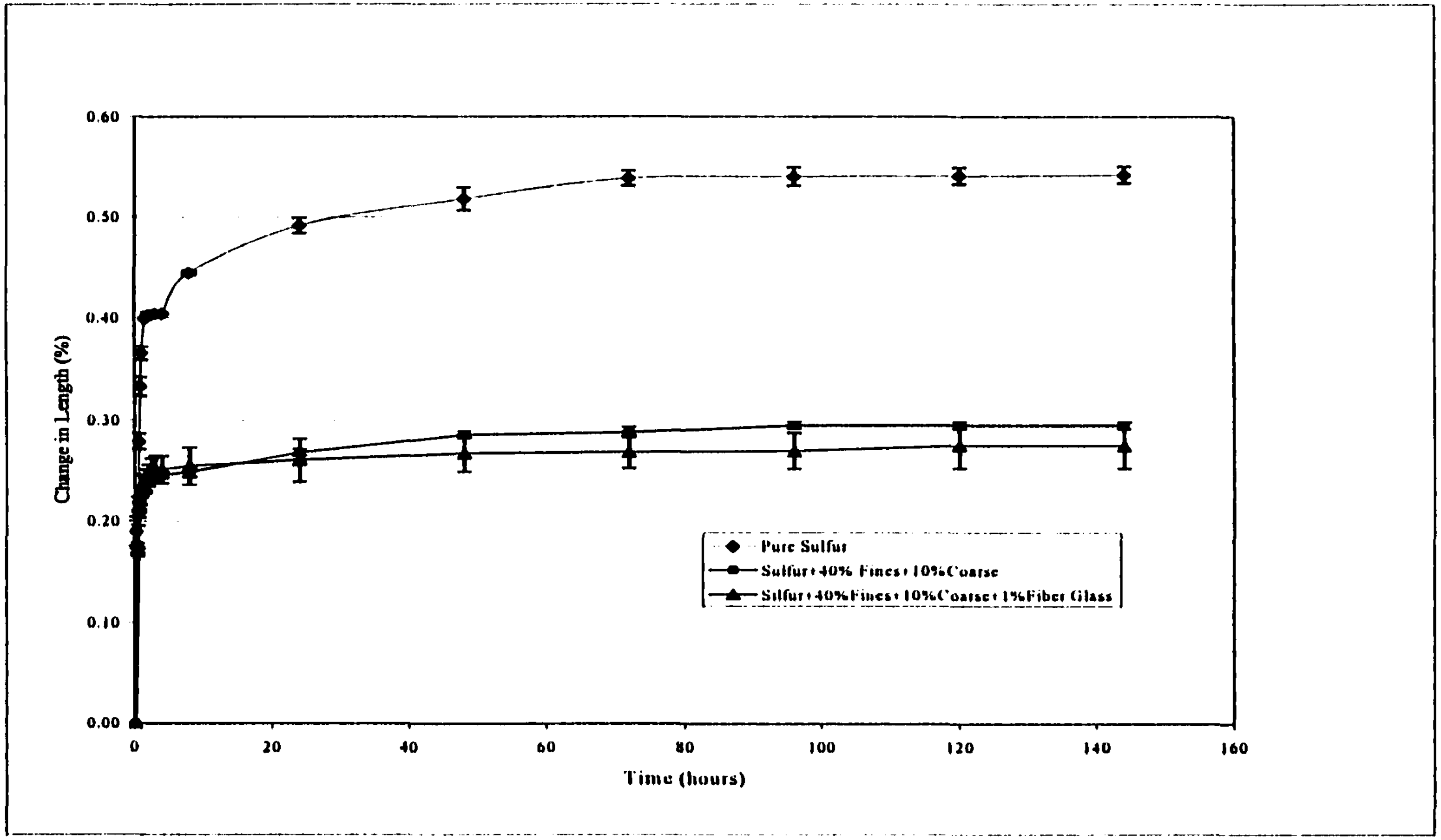


Figure 21: Change in length with time for pure sulfur and sulfur mortar

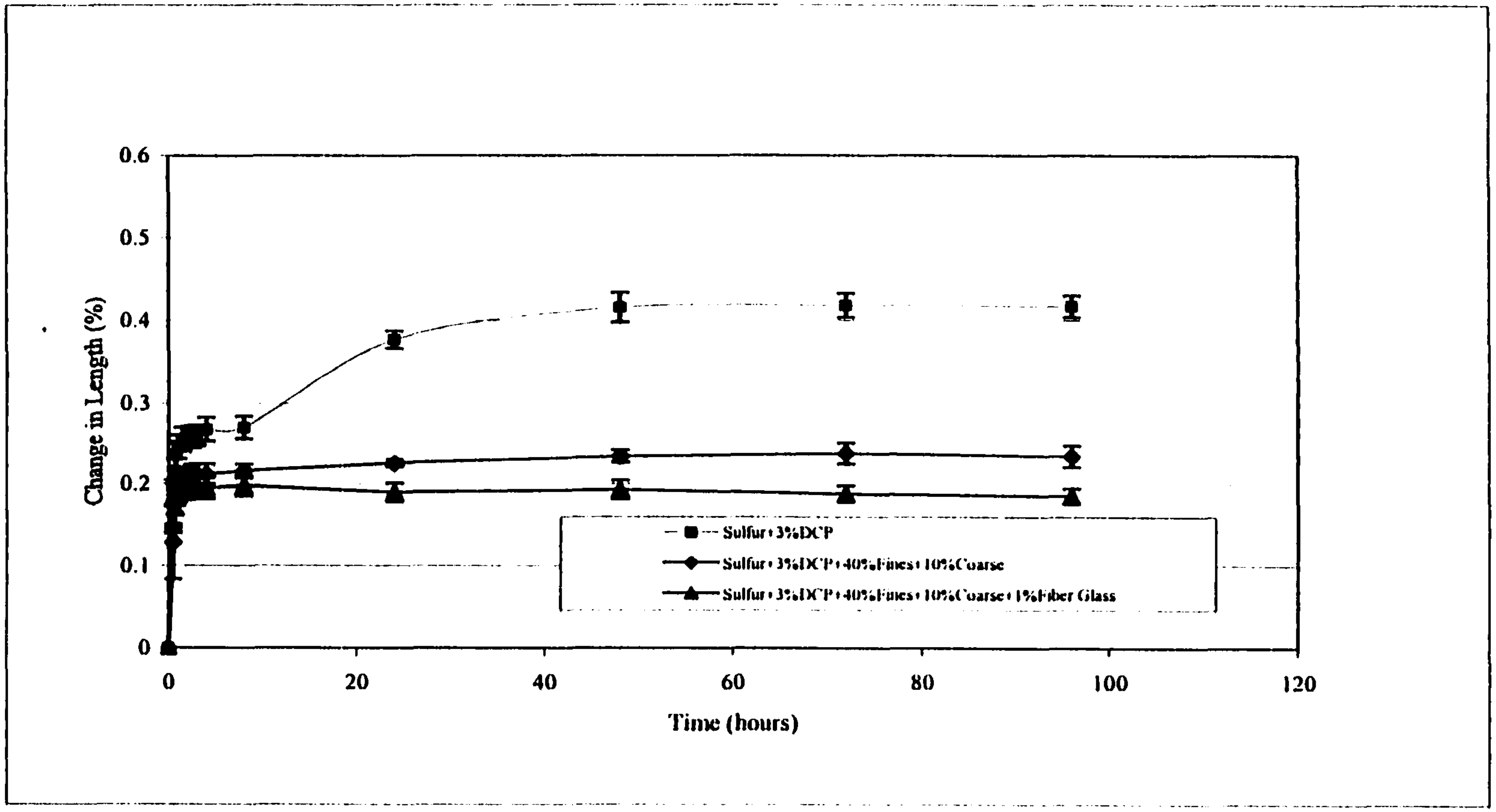


Figure 22: Change in length with time for 3% modified sulfur and modified sulfur mortar

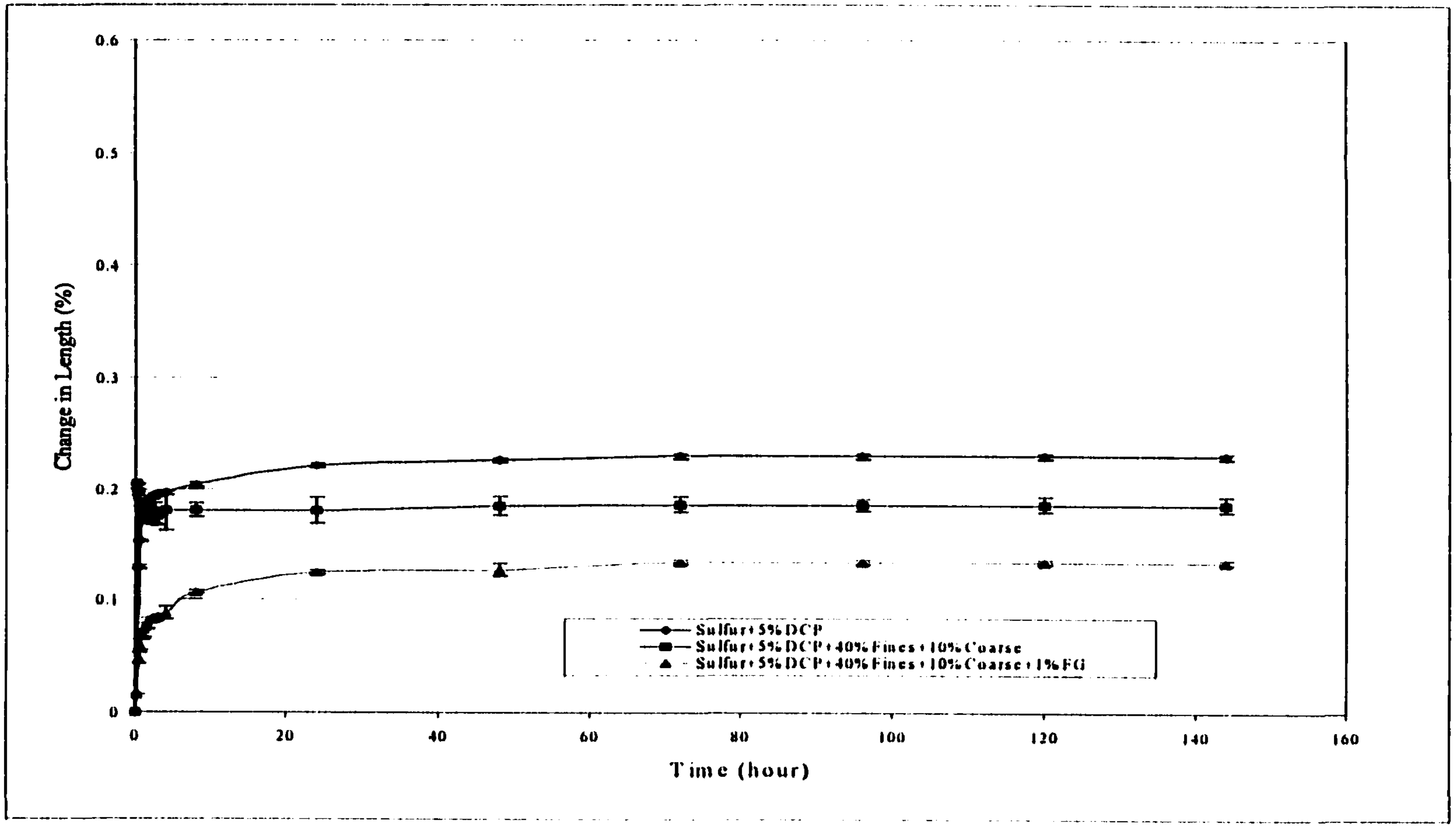


Figure 23: Change in length with time for 5% modified sulfur and modified sulfur mortar

Sulfur Film Thickness

For application and illustration purposes, the different mixtures used were poured inside a 2 foot diameter pipe while the pipe was being rotated, and the thickness of the film obtained was measured for all mixtures used. The importance of this step was to determine the film thickness in the case of free pouring of the material from an outlet inside the pipe. The test was performed by pouring the mortar inside the pipe from an outlet, then the film obtained was chipped and its thickness was measured using a Vernier Caliber. Testing results are given in Table 27.

The general pattern, although not clear, was that the film thickness would increase with increasing the aggregate content. The interaction between the viscosity and the aggregate content may have affected the test results. A lot of variables were involved in preparation of the mixtures such as mixing time, mixing temperature, and percent of modification obtained may have precluded obtaining completely accurate results.

Acid Penetration

The samples used for the study of the effectiveness of DCP modified sulfur as a barrier to acid were made by casting uniform mortar layers of 1/8" over conditioned concrete bricks. After solidification of the mortar, a plastic container with an area of 7.56 in.^{2} was cut from the bottom and was sealed to the solidified mortar surface with a silicon compound. A 10% sulfuric acid solution was placed in the container and with direct contact with the DCP modified sulfur surface. 10% sulfuric acid was used to simulate sulfuric acid formed in the sewage systems. No noticeable reaction occurred on the substrate covered with DCP modified sulfur.

Table 27: Film thickness (inch) when sulfur is poured inside a 2 feet diameter pipe

	0% DCP					
	Sulfur	10%	20%	30%	40%	40%+10%
Trial 1	0.0160	0.0400	0.0310	0.0240	0.0270	0.0330
Trial 2	0.0180	0.0390	0.0320	0.0250	0.0280	0.0320
Trial 3	0.0190	0.0360	0.0360	0.0300	0.0320	0.0350
Trial 4	0.0200	0.0360	0.0380	0.0320	0.0300	0.0360
Trial 5	0.0200	0.0390	0.0350	0.0300	0.0320	0.0340
Average Thickness:	0.0186	0.0380	0.0344	0.0282	0.0298	0.0340
Standard Deviation:	0.0017	0.0019	0.0029	0.0035	0.0023	0.0016
	3% DCP					
	Sulfur	10%	20%	30%	40%	40%+10%
Trial 1	0.0400	0.0280	0.0420	0.0280	0.0460	0.0480
Trial 2	0.0420	0.0290	0.0450	0.0330	0.0500	0.0420
Trial 3	0.0420	0.0290	0.0470	0.0370	0.0460	0.0470
Trial 4	0.0410	0.0270	0.0470	0.0350	0.0487	0.0430
Trial 5	0.0430	0.0280	0.0450	0.0360	0.0500	0.0460
Average Thickness:	0.0416	0.0282	0.0452	0.0338	0.0481	0.0452
Standard Deviation:	0.0011	0.0008	0.0020	0.0036	0.0020	0.0026
	5% DCP					
	Sulfur	10%	20%	30%	40%	40%+10%
Trial 1	0.0250	0.0300	0.0350	0.0400	0.0450	0.0500
Trial 2	0.0260	0.0290	0.0350	0.0400	0.0420	0.0520
Trial 3	0.0270	0.0300	0.0380	0.0400	0.0450	0.0500
Trial 4	0.0250	0.0280	0.0360	0.0390	0.0430	0.0480
Trial 5	0.0260	0.0290	0.0350	0.0410	0.0420	0.0460
Average Thickness:	0.0258	0.0292	0.0358	0.0400	0.0434	0.0492
Standard Deviation:	0.0008	0.0008	0.0013	0.0007	0.0015	0.0023

Summary and Conclusion of Laboratory Testing

The objectives of the laboratory-testing program of the research were to evaluate the effects of polymer modification of sulfur mortar, its workability and ease of use when lining concrete sewer pipes. Additives studied include Dicyclopentadiene (3 and 5 % by weight) and fiberglass. Engineering properties studied in this phase include compressive strength effect against aggregate content, bond strength, viscosity characteristics, resistance to sulfuric acid, and shrinkage characteristics

In summary, the optimum DCP content from results of compressive strength testing performed was 3% by weight of the sulfur. In general, modification of sulfur mortar with DCP resulted in improved compressive strength and less shrinkage. In addition, DCP modified sulfur is resistant to sulfuric acid. Fiberglass reinforcement is a beneficial additive for sulfur mortars, since it eliminates loss of compressive strength with time that occurred with all materials.

Modified sulfur mortars are less susceptible to cracking due to shrinkage that will occur during cooling of regular sulfur mortar. Laboratory test results show that modified sulfur mortars experienced less shrinkage than that of sulfur mortars. As a result, sulfur and modified sulfur mortars used gave similar or even at some instances better compressive strength and provided superior sulfuric acid resistance than that of Portland cement concrete.

MANUFACTURING PROCESS OF SULFUR LINED CONCRETE PIPE

Introduction

This portion of the research presents the equipment, manufacturing processes, trials for lining the concrete sewer pipe and lessons learned in the lining process. Also, the intent of this portion of the study was to produce a full-scale sulfur mortar lined concrete sewer pipe to be used on a commercial scale.

Laboratory evaluation of materials to be used was performed in the previous parts of this research. Materials evaluated included sulfur mortar, polymer (Dicyclopentadiene, DCP) modified sulfur mortar, fiberglass reinforced sulfur mortar, and fiberglass reinforced DCP-modified sulfur mortar. In the previous parts of the research, it was found that modifying the sulfur would result in better physical and engineering properties of the material. The optimum DCP content from results of testing was determined as 3% by weight of the sulfur.

In general, modification of sulfur mortar with DCP resulted in better strength and less shrinkage. DCP modified sulfur is resistant to sulfuric acid. Fiberglass reinforcement is applicable for use with sulfur mortars since it eliminates loss of strength with time that occurred with all materials used.

Progress of Processes of Manufacturing

In a sanitary sewer, it is critical to minimize infiltration and exfiltration through the pipe. For these reasons different jointing procedures are used. This project was initially directed toward a rigid joint system that will produce mainly a pipeline system with fixed or rigid type of joint. The rigid joint system has several disadvantages including: difficulty of installation and alignment of the trench, sensitivity of the system to breakage due to deflection or movement, and difficulty in obtaining continuous and smooth flow line. This approach was abandoned and a flexible jointing system was adopted due to the need for flexibility of the pipeline to accommodate lateral deflection and the need for ease of installation.

In the previous parts of this research, it was illustrated that lining can be obtained using a heated form that is placed inside the pipes, sulfur mortar can then be poured in the annular space. The pipe size used in the previous approach was 1-foot diameter pipe and this required small volumes of sulfur mortar. The trials were successful, but when the form or mold was heated, the sulfur solidified before filling the annular space between the pipe and the mold. Electricity was used to heat the mold; this produced super heated spots around the heat sources, and some locations of the mold where not hot at all. Also, when trials were made with larger pipe diameter, the volume of sulfur mortar used was much greater than the volume used with the 1-foot diameter pipe, this resulted in severe cracking of the liner.

Based on the results of testing and from careful consideration of the parameters involved in the manufacturing process, the following objectives had to be changed:

1. Changing the joint system from a fixed restrained system to a flexible jointing system to facilitate installation and produce an acceptable pipe that can be used by the market or the industry;

2. Increasing the volume of sulfur will result in more shrinkage. More shrinkage will produce more cracking, thus; modification and reinforcing the sulfur was necessary in order to produce a non-cracked section; and
3. Using a melting and pouring system that will provide homogeneous temperature that will aid in providing better mixtures and will produce a homogeneous and continuous liner.

Selection of Pipe, Joint, and Material

The next step of the research at this point was to determine the most popular size of concrete sewer pipe used in the market and study the feasibility of lining the pipe at laboratory scale. After research of the concrete sewer pipe market, it was determined that a 2-foot diameter, 8-foot length pipe can be considered one of the most popular sizes used, but the 8-foot length prohibits handling the pipe in the laboratory easily.

Iowa Concrete Products, Inc. (CRETEX) in Des Moines, Iowa donated the representative pipe for this research provided that the supply will be in 6-foot lengths for ease in the handling process. The original pipe selected had bell and spigot joints. A rubber O-ring is commonly used as the sealant for this type of pipe. The pipe and joints dimensions are illustrated in Figures 24 and 25 below.

The second step of this research involved determination of the optimum joint configuration to produce a pipe that is acceptable in the market, and at the same time meets the standard specifications to produce a watertight joint. After consideration of several alternatives available in the market such as rubber O-ring and several sealing procedures including the rigid pipe system, it was decided to use Forsheda's, Inc. single offset joint due to proven performance and popularity that will facilitate adoption of the manufactured pipe. This type of joint employs a rubber gasket with a special design in order to perform as the sealant. The new adopted joints dimensions are illustrated in Figures 26 and 27 below. The production of this type of joint required new casting of the whole joint, i.e. producing a new bell and a new spigot.

Also, in the process of this research, an essential part was obtaining silica-based aggregate and sulfur for commercial scale applications. The initial trials involved purchasing 50-lbs bags of ready-to-use aggregate. The price of the material ranged from $15 to $25 per bag. This cost was determined to be expensive considering the amounts of aggregate to be used in the manufacturing process. In addition, laboratory scale suppliers would provide the sulfur for a cost of $7 per pound. After contacting several quarries and sulfur suppliers, the materials were obtained for the research for minimal prices.

The cost of unclean, 90% passing sieve # 200 silica sump sand was approximately $5/Ton plus shipping and handling and this would require sieving of the material to conform to a cleaner aggregate. The aggregate was purchased from Spencer Quarries in Spencer, South Dakota. The sulfur was purchased from International Sulfur, Inc. in Texas for $850/2500 lb., and this cost includes shipping and handling of the material delivered to Ames, Iowa.

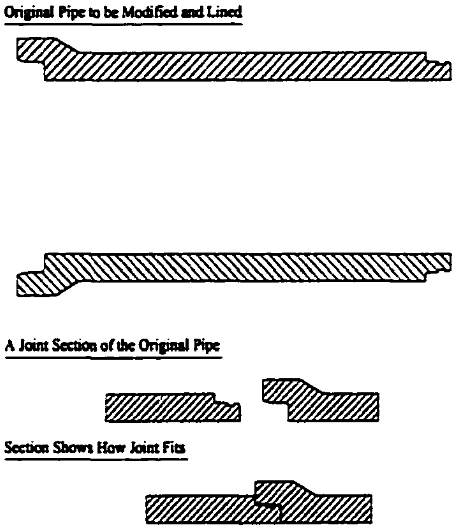


Figure 24: General geometry of the pipe and joint to be modified (Iowa Concrete Products, 1998)

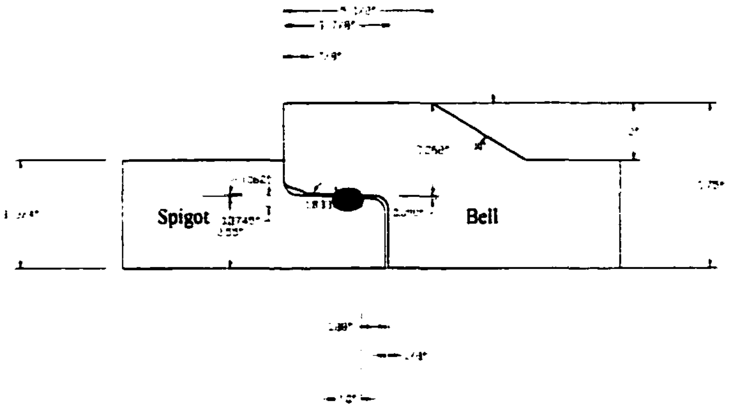
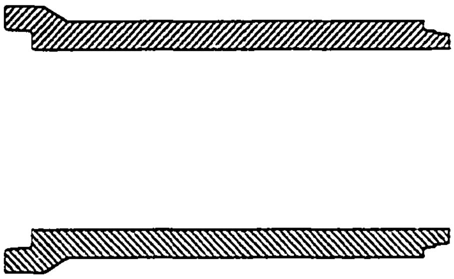


Figure 25: Dimensions of the joint to be modified (Iowa Concrete Products, 1998)



A Joint Section of the Modified Pipe

Section Shows How the New Joint Fits

Figure 26: Geometry of the pipe and joint to be manufactured

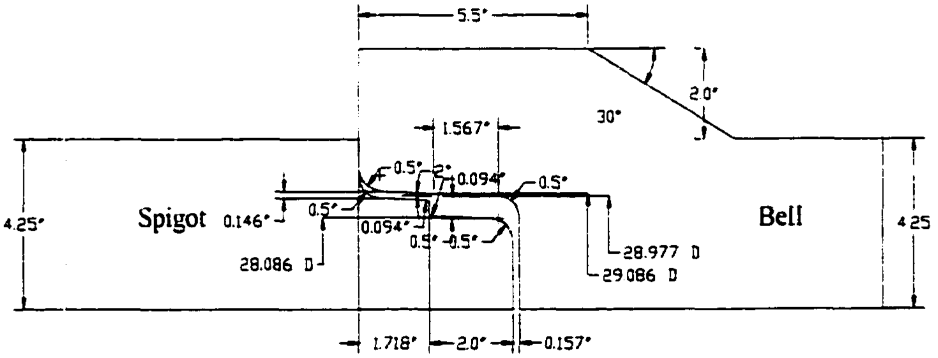


Figure 27: Dimensions of the single-offset joint to be manufactured (Forsheda, Inc. 1998)

Processes of Manufacturing

General

In general, the concept of lining an existing concrete sewer pipe involved two steps, forming the joints and forming the core of the pipe. Several processes were introduced to manufacture a sulfur-lined concrete sewer pipe. These processes included a variety of equipment and sub-processes and are described below.

One process involved setting the pipe vertically, allowing a heated form to slide while the sulfur is being poured around the form. While the form is being sledded, the sulfur will be given enough time to solidify. This process was abandoned due to the fact that a significant number of controls were required for the process such as pouring discharge, that will vary from one pipe size to another, increasing speed, temperature control and the temperature of the form. In addition, complicated mechanisms were required to control pouring and raising the form at the same time.

Another process was introduced and evaluated. This process was promising due to the minimum controls required during the process of manufacturing. The idea in this process was to employ some forms that will be installed in and around the pipe in order to obtain an annular space for the portion of the pipe to be lined. Then, using an inlet, the molten sulfur mortar will be poured into the annular space and the mortar allowed to cool down. Then the molds or forms are stripped. A more detailed description is given in the following sections. This process is identified as Process I, and will be described in the following sections.

A third process was proposed using forms and rotation of the pipe. This process was introduced due to reasons including cracking that occurred after completion of lining in the Process I, and the presence of fiberglass reinforcement plugged the mortar inlet. The process involved forming the joints using a set of pre-manufactured molds for the joints. Then using a specially designed tank, the molten mortar will be applied on the core of the pipe while rotating it, the rotation will provide a fast cooling mechanism for the mortar. This will produce a quenching process through which the material cools fast and this will prevent further cracking. A more detailed description is given in the following sections. This process will be identified Process II and will be used in the following sections.

Melting and Pouring Tanks

In a previous part of this research, it was determined that a liner of ¼" thickness will provide satisfactory performance regarding bond strength and acid resistance for penetration. For a production line that involves lining three 8-foot diameter pipes, the volume of sulfur required to line one pipe with a ¼" thick liner was estimated to be around 3 ½ ft^3. A tank of at least 12 ft^3 volume is required considering a mixing mechanism needed in the mix to assure obtaining a homogeneous mix. The volume required for providing a lining for one 144" diameter pipe is approximately 20 ft^3.

The sizing of the melting tank was based on the ability to provide material to manufacture two of the largest pipes available in the market, and to provide as much as of materials for the prototype selected for this project. The melting tank was designed to handle at least ten 2-foot diameter pipes with a volume of approximately 40 ft^3.

Providing homogeneous temperature to melt the sulfur and to assure efficient polymer modification of the sulfur was an essential part of this project. One of the most successful processes used to provide homogeneous temperature for heating purposes is using a hot oil bath to provide for needed temperature. The required melting, reaction and pouring temperatures ranged from 220 °F to 290 °F, and temperatures above 310 °F will result in burning of the sulfur.

Manufacturing two tanks, one inside the other, provided these temperatures. A large tank to provide the hot oil bath and a small tank to be installed in the larger tank to provide for melting of the sulfur. Dimensions of the small tank were 4x4x2.5' (40 ft^3), and dimensions of the large tank were 5x5x4'. A tube burner that zigzag's in the oil was used to provide the temperature for the oil that will be transferred the to the sulfur. A schematic of the tank is shown in Figure 28 below.

The heat transfer medium used was non-detergent grade oil to keep up with a safer environment. CITGO's Pacemaker oil was used for a cost of approximately $275/50-gallons drum as the heat transfer medium. LP gas was used to as the energy source for the burners in this project. Heat was provided for the oil using a heavy-duty burner that provide 500,000 Btu and this burner was manufactured by the Walling Company.

The heating tanks were provided with a digital temperature indicator and controllers, a thermocouple sensitive up to 1 °F, air power fan, and gas and air controllers. The tank system was provided with two outlet valves, a ball valve which is used to empty the oil for transporting and cleaning purposes, the other valve was a gate valve that is used to pour the hot molten mortar out from the melting tank for the pouring process.

The outlet valve was sized to be 2 inches valve that can handle a temperature up to 280 °F. The valve's gaskets were worn out after three to four pouring trials and it needed to be unplugged after each trial. The gate valve size were then changed to 4 inches instead of 2 inches, and a 4 inches valve was produced for this process.

The inner melting tank was provided with a mixer that is driven by ¾ -HP electrical motor. The electrical motor was used to drive two-1.75' propellers in order to obtain homogeneous mixing during the modification process. Also, a pouring tank was designed during this process. The mechanisms involved in the pouring tank are illustrated in Figure 29.

The pouring tank was designed with a gate valve that is operated using a remote handle for safe opening and closing. The tank performed well except that a heating mechanism would be required to elevate the temperature of the tank and valve for easy operation. A regular 120,000 Btu gas burner was used for this purpose. A tank with 2'x2'x3.5' was designed for this purpose. The tank's dimensions and mechanism are illustrated in Figure 30 below.

The total cost of the melting tanks, pouring tanks, with the accompanied burners and controllers was approximately $16,000 with more than $10,000 employed in burners and controllers.

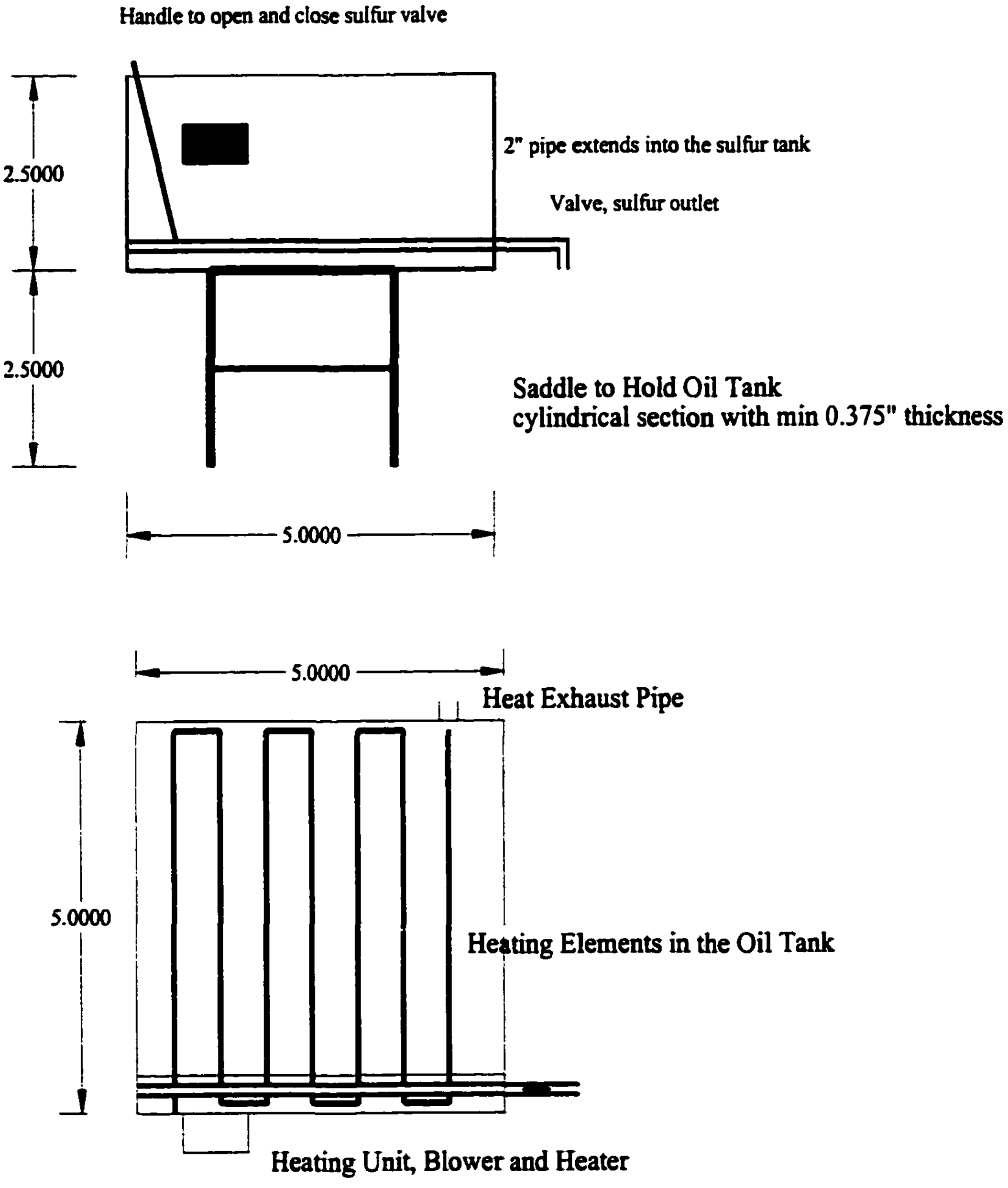


Figure 28: Schematic of the heating tank

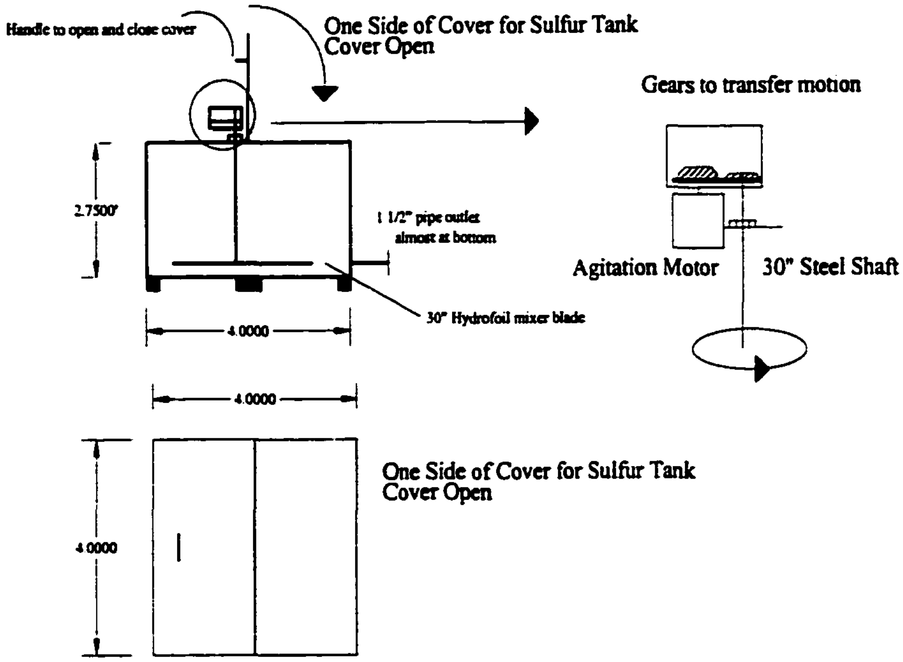


Figure 29: Dimensions and mixing mechanism of the melting tank

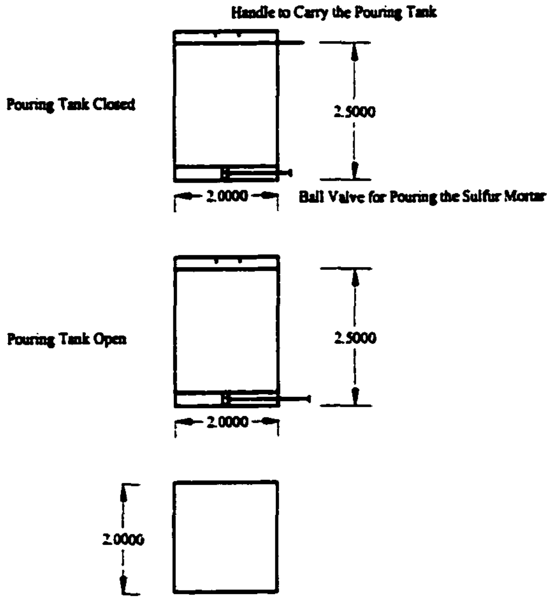


Figure 30: Dimensions of schematic of the pouring tank

Manufacturing Process I

Pouring Molds

As mentioned in the previous section, the first process involved forming an annular space between the pipe and a set of molds, after that, the molds are heated, then the annular space is filled with molten sulfur mortar. Initial experimenting of the molten mortars indicated that using larger volumes of the material would result in more shrinkage. The materials also will bond with both steel and concrete. This necessitated that a release mechanism should be applied for stripping the molds out of the pipe after application of the liner.

For the previous reasons, the research in this stage was directed toward obtaining a solution for the bonding problem before the application starts. The solution was to use a collapsing mechanism that will be supplied with the molds and the inner core. By contacting several pipe equipment manufacturers, it was decided to manufacture the molds and the inner core at Glenn Machine and Foundry in Boone, Iowa. This company provided the flexibility of the production line and at the same time they were one of the few companies that manufactured molds for the single offset joint configuration.

The molds were manufactured in five pieces according to the dimensions shown in Figures 31 through 34 and they are:

1. Inner collapsible core to provide an inside lining of ¼" or a diameter of 23 inches.
2. A mold to form the spigot with the single offset joint configuration;
3. A mold to from the bell with the single offset joint configuration;
4. An outer jacket to form and hold the spigot mold inside and around the pipe; and

5. An outer jacket to form and hold the bell mold inside and around the pipe.

The collapsible mold employs a crack that opens and closes using a set of screws that are operated by a handle. When the screws are tightened, the crack is sealed (larger diameter is obtained) and when the screws are loosened, the crack is opened (smaller diameter is obtained). Also, in order to ensure a continuous flow of the molten sulfur mortar, a heating mechanism had to be provided for the inner core. A 500,000 Btu burner that was supplied by the Walling Company was also installed inside the collapsible core. The burner was provided with controls to increase and decrease the temperature manually. The cost of the molds was approximately $7,000, with 60% of the cost employed in manufacturing the collapsible mold.

Operation of Manufacturing Process I

The initial trial in this process was to line the pipe using a polymer modified sulfur mortar compound donated for the project. Initial trials were to be made using this material in order to minimize cost of the original material to be used and to experiment using the system. The assembly of the mold system was difficult due to the heavy weights of the molds.

The molds were manufactured using typical dimensions provided by the pipe manufacturer. The dimensions did not match the actual manufactured pipe and this produced some difficulties in the assembly process. A lubricant was applied on the inner surface of the molds in order to facilitate stripping molds. The molds were assembled around the pipe and initial examination of the assembly showed that there were some spaces present between the pipe and molds. The spaces or voids needed to be sealed to prevent leaking of the mortar.

The next step was to form the pouring inlets in the assembly. Three 1 ½ inches holes were drilled in the pipe body using a hammer drill. Multiple holes or inlets were used to provide extra inlets in case any of the inlets became plugged and at the same time to provide an indicator that the annular space was full.

After melting the mortar, assembling the molds around the pipe and heating the core, the mortar was transferred into the pre-heated pouring tank, then using a funnel, the material was poured into the annular space. Pouring was continued until the mortar started flowing out of the inlet holes and to compensate for the loss in volume due to shrinkage of sulfur. Figure 35 illustrates the assembled pipe and molds system with the various tank components and gives a general description of the operation of this process. Figure 36 illustrates the assembled pipe and the molds system.

After completion of the pouring operation, the burners were turned off and the molds systems were stripped after approximately 12 hours. The collapsible mold and the joint molds were removed from inside and around the pipe. The lining was complete in terms of the volume of mortar applied, but the material experienced severe cracking that increased as the time passed by.

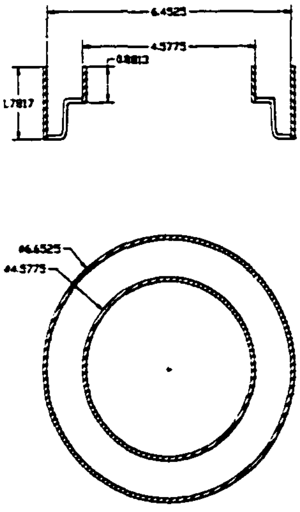


Figure 31: Dimensions and general parts involved in manufacturing the Bell

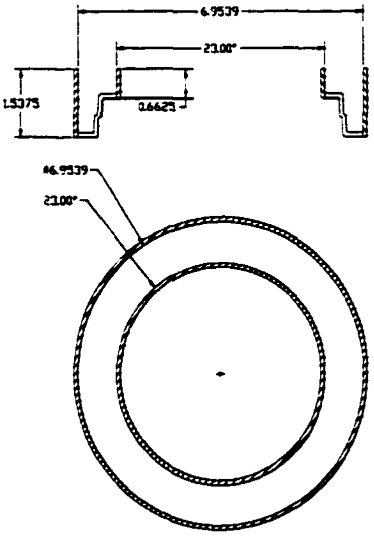


Figure 32: Dimensions and general parts involved in manufacturing the Spigot

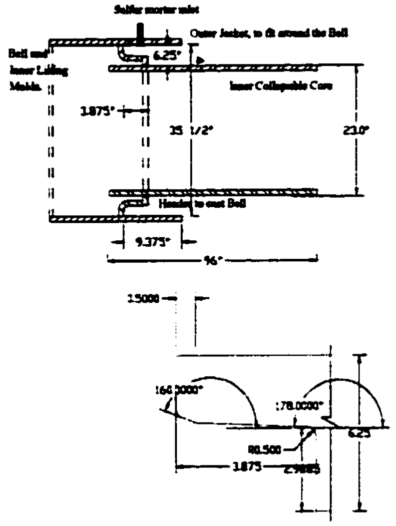


Figure 33: General configuration of the Bell mold

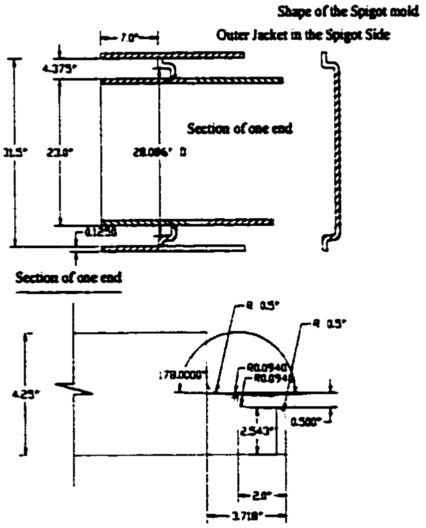


Figure 34: General configuration of the Spigot mold

The Overall Lining System

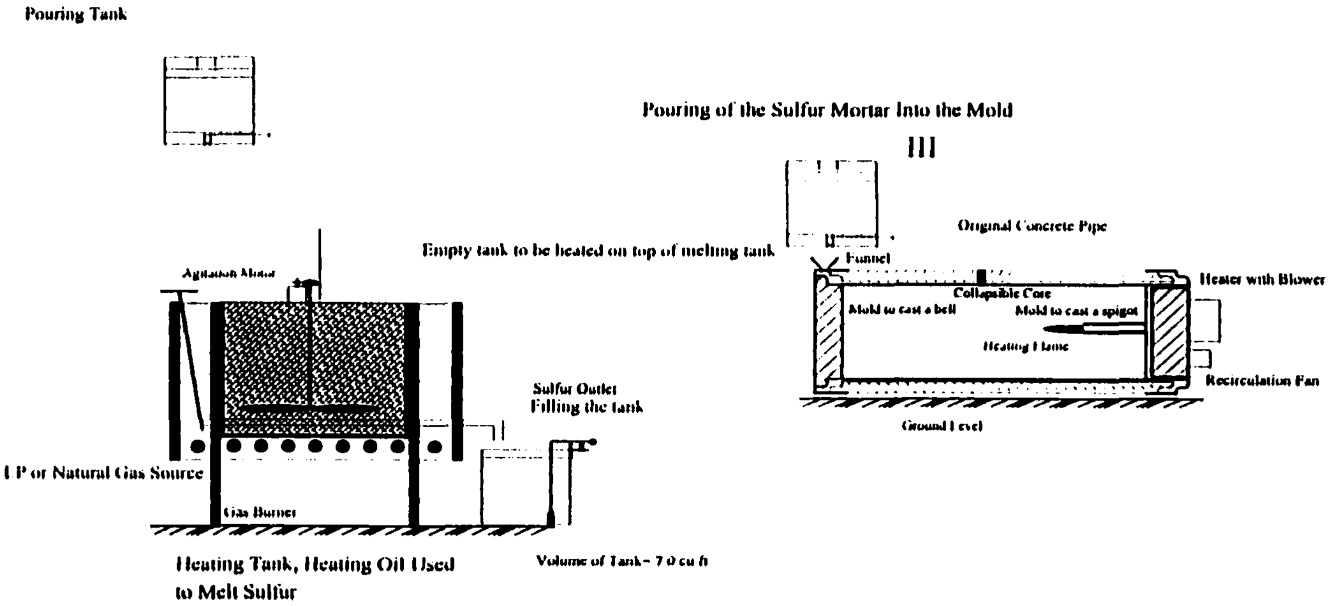


Figure 35: General schematic of the overall lining and operation of Process I

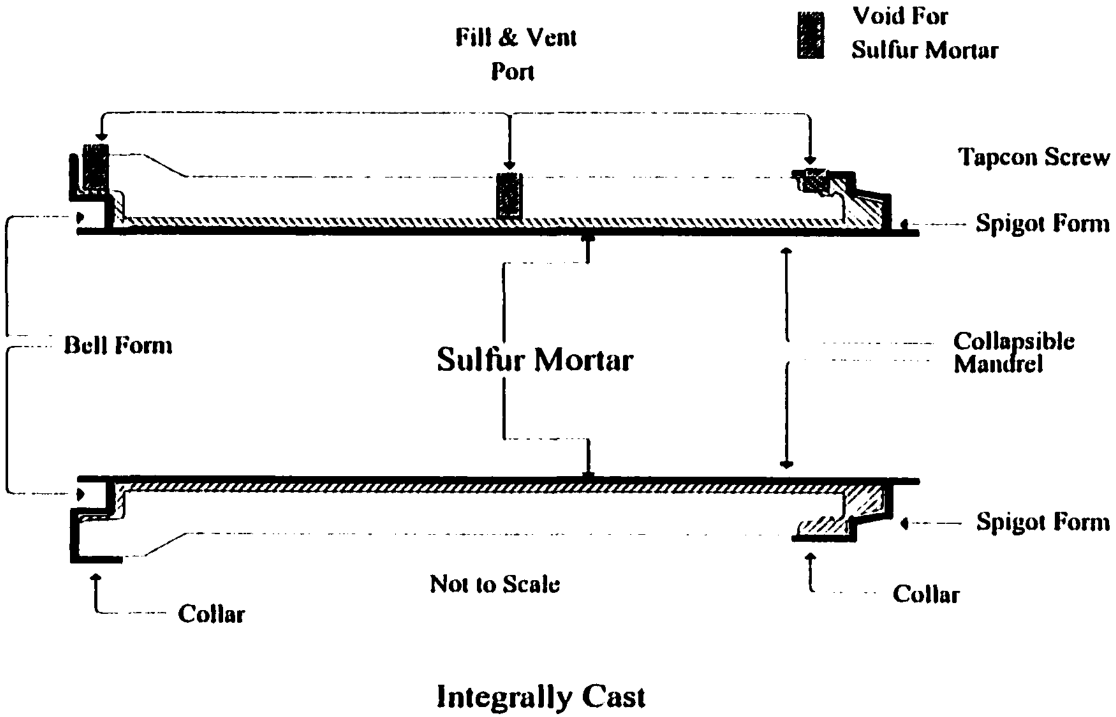


Figure 36: The assembled pipe and mold system

Other trials to line pipes were performed using non-modified sulfur mortar. In the first trial, approximately 75% lining was obtained due to leaks that occurred in the assembly. Also this resulted in delays that resulted in the sulfur to cool down in the pouring tank and solidify. Also, after examining the lining, cracking occurred in all sections of the pipe, and the most severe cracking occurred in thicker sections of the lining.

The decision then was directed toward reducing potential cracking. This was atteapted by manufacturing the thickest section available along the pipe, i.e. the spigot. The spigot was formed using a mold and a plastic form to provide the required diameter. Several trials were made to manufacture a spigot using different materials and procedures, such as pouring the material in layers and allowing it to solidify, applying steel wire reinforcement, applying steel mesh reinforcement, polymer modification of the sulfur, and fiberglass reinforcement of the mortar. The only successful mixture that enabled the production of a joint with no cracking was using 3% DCP modified sulfur mortar (40% fine aggregate + 10% coarse aggregate) with 1% fiberglass reinforcement.

The successful mixture then was prepared to line a full-scale pipe. The molds and pipe were then assembled; the inner core's temperature was elevated to the pouring temperature. When the material was poured in the first inlet of the pipe, the first inlet was plugged due to the accumulation of fiberglass in the hole. The same problem occurred in the other holes. Less than 50% of the pipe was lined using this material.

This overall process involved several advantages and disadvantages. The advantages included less control needed in the pouring process, the smoothness of the liner and matching to the molds surfaces, full lining of the pipe was performed in one step, and speed of the process. However, this process seemed to have more disadvantages than advantages. Leaking of the mortar between the molds and pipe was an important issue; rubber gaskets were not sound enough to stop the leak. The process also required extra manpower to handle the heavy molds and heavy pouring tank. Also, plugging the pouring inlets with fiberglass with more viscous mixtures was a serious problem. Figures 37 through 47 illustrate the various failed trials and the actual manufactured components in this process.

Figure 37: Photo illustrating the melting tank and heating unit

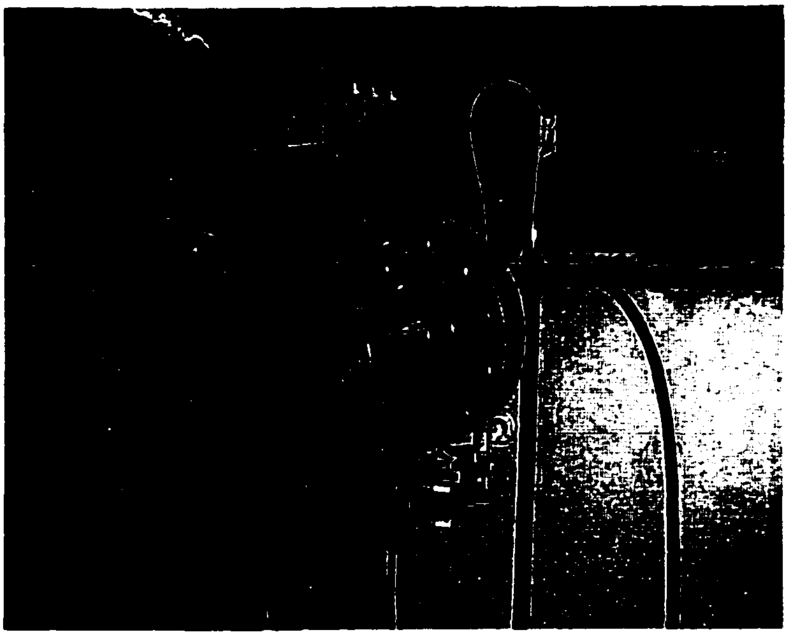


Figure 38: Photo illustrating a closer look at the heating unit for the heating tank

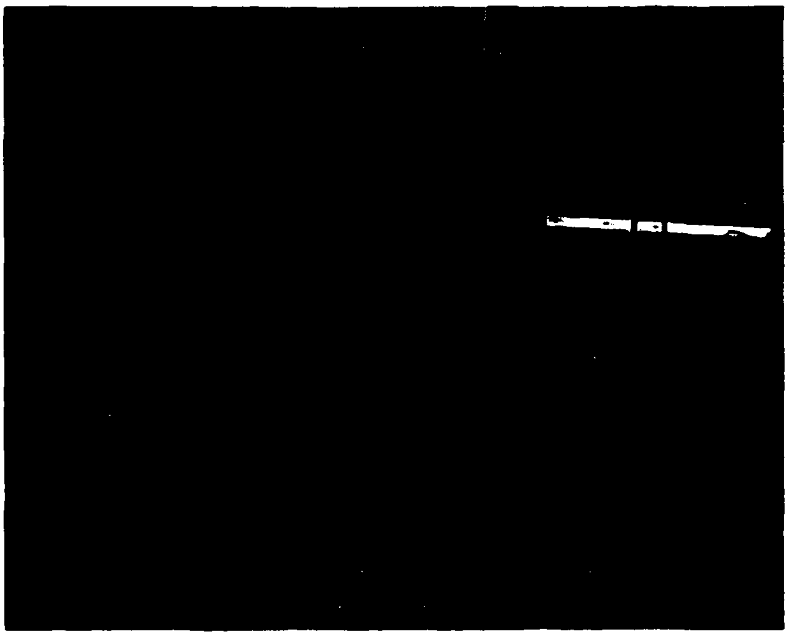


Figure 39: Photo illustrating the pouring tank

Figure 40: Photo illustrating the oil used in providing the heating medium

Figure 41: Photo illustrating the Bell jacket, the collapsible core and the heating unit

Figure 42: Photo illustrating the collapsing mechanism of the core

Figure 43: Photo illustrating trials of reinforcement of the Bell

Figure 44: Photo illustrating trials of reinforcement of the Bell

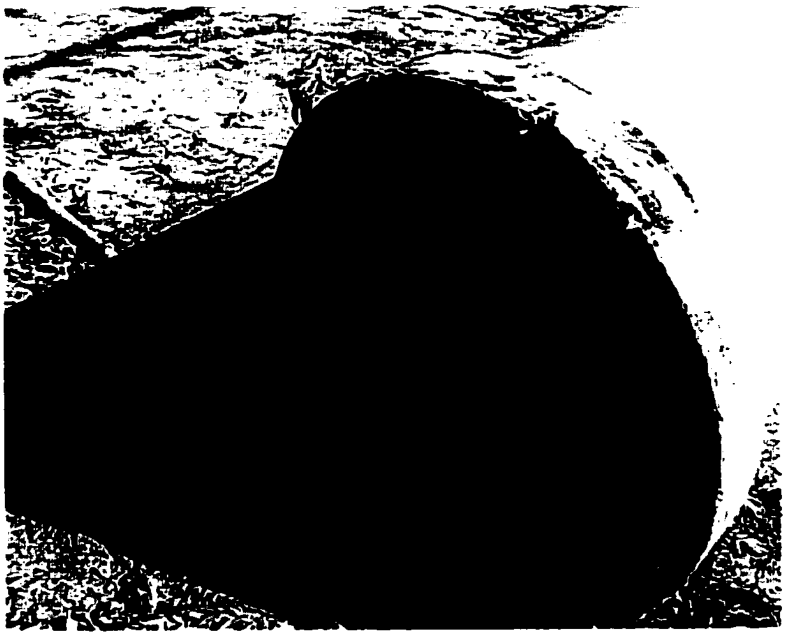


Figure 45: Photo illustrating a failed trial of lining using process I

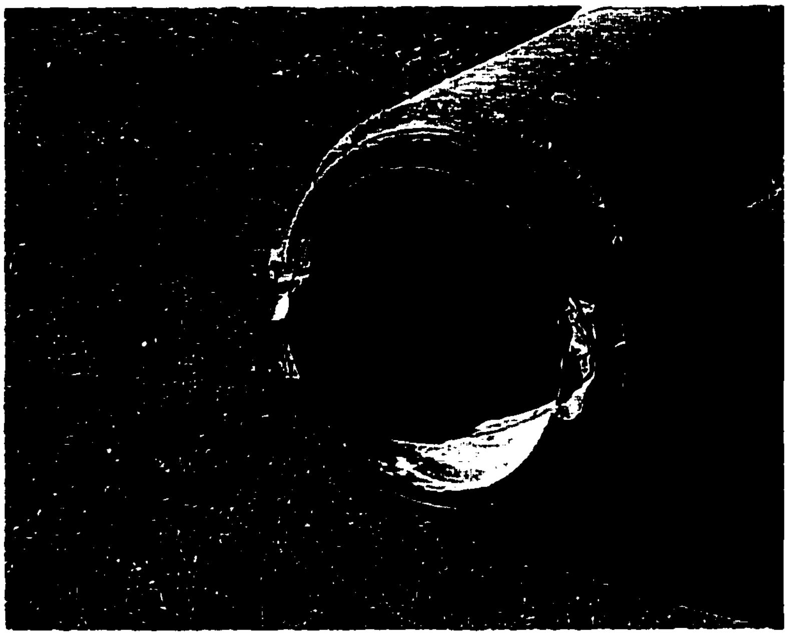


Figure 46: Photo illustrating a failed trial of lining using process I

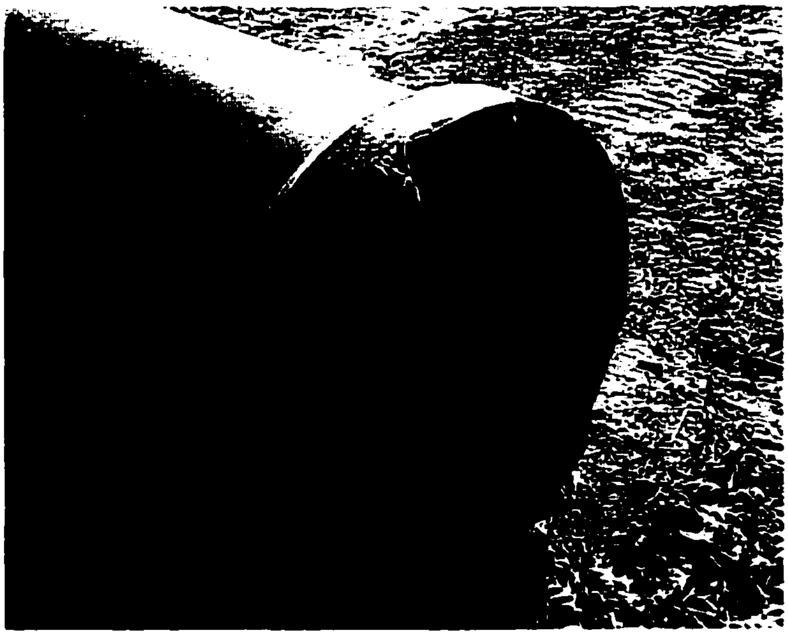


Figure 47: Photo illustrating a failed trial of lining using process I

Manufacturing Process II

Pouring Tank and Frame

At this stage and since a lot of problems occurred during the lining using manufacturing process I, the whole procedure was directed toward using another method to line the pipe. Another approach was proposed, to utilize using a fast cooling process. The process was implemented by pouring the sulfur mortar while rotating the pipe, and at the same time use the joint molds that were used in manufacturing process I.

The process was implemented by designing and manufacturing a heating tank with a length equal to the pipe length. Mortar outlets were manufactured using two sliding plates with matching holes. One plate was allowed to slide in relative to the other, and when the holes meet, the sulfur mortar started flowing. Electrical strip heating units were provided along the sides of tank to maintain and provide high temperature during the pouring process. Also, two valves were installed at each end of the tank to pour the sulfur mortar while forming the joints. The outlet valves were also heated using strip-heating units to prevent plugging of the valves and to assure continuous flow of the mortar.

The rotation mechanism was obtained by manufacturing a steel frame with heavy-duty rubber wheels to rotate and hold the pipe. The frame was manufactured to support the pouring tank, and at the same time support and rotate the pipe. The frame was designed in accordance to the Load and Resistance Factor Design (LRFD). The rotation was obtained using a hydraulic pump and motor that operates a rubber tire that is kept in contact with the exterior wall of the pipe. Figures 47 and 48 present schematics of the equipment, dimensions used and the assembled pipe in this procedure.

Operation of Manufacturing Process II

At this stage, and since a fast sulfur cooling mechanism was used, the process was directed toward using non-modified sulfur mortars to line the pipe. In addition, the Dicyclopentadiene (DCP) modifier was a highly flammable material and using it resulted in a malodorous smell. This led to the use of unmodified sulfur mortar that contained silica crusher fines as the base liner to line the concrete pipe.

In general, manufacturing process II required less preparation than that of the previous process, although more manpower was required to operate this process. Also, in this process, the use of unmodified sulfur was proposed to line the pipe. Initial preparation of this system required installation of the joint molds; the joints were installed and fixed on the original pipe using Tapcon screws. An annular space was provided between the pipe and the molds to provide for the new formed joints. In the first stage the joints were poured using a pot while rotating the pipe. Fiberglass reinforcement was applied as the material is poured into the joints.

After casting the joints, hot sulfur mortar material was poured into the hot pouring tank. The pipe then was allowed to rotate and then the sulfur mortar was poured. The sulfur mortar solidified immediately when contacted the pipe, this process was continued until the whole core was lined. The process of preparation and lining took about 30 minutes. Visual inspection of the produced lining indicated that it did not experience any cracking, which is an indication of less shrinkage experienced by the sulfur mortar. Also, visual inspection of the produced lining appeared to be smoother and posses less voids than the concrete pipe surface, this is an indication that the sulfur lining will posses less permeability than concrete.

The Tapcon screws were then cut and the molds were stripped out, the presence of exposed steel screws may develop a potential weak plane that may be attacked by sulfuric acid, this can be prevented by applying a sulfur cover on the exposed portion of the screw.

As a result, manufacturing Process II was adopted successfully in manufacturing a concrete sewer pipe lined with sulfur mortar. Figures 48 through 59 shows schematics of the design and photos that were taken for the manufactured system and the lined pipe.

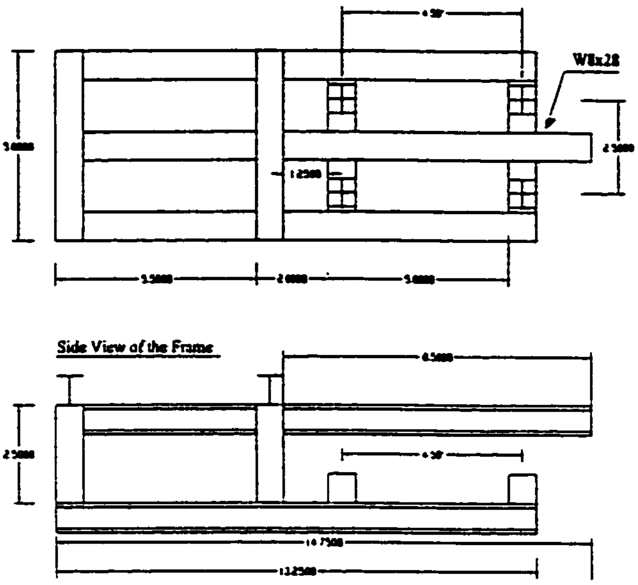
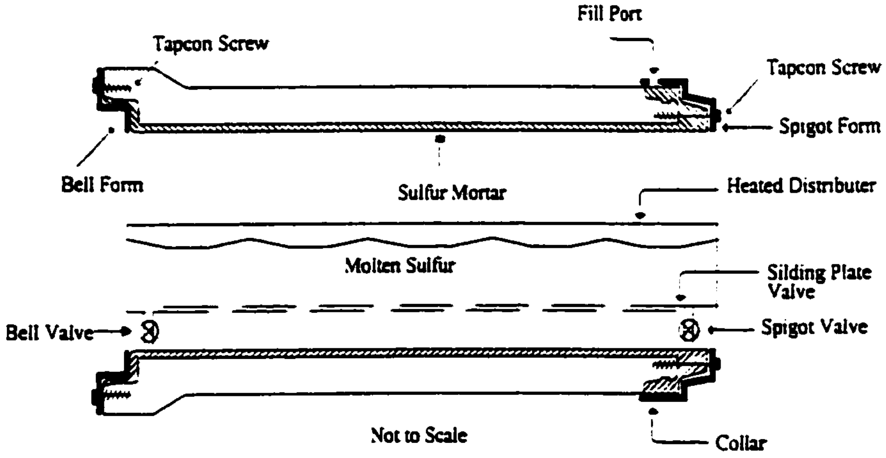


Figure 48: Dimensions and general shape of the steel frame used in Process II



Roll Cast

Figure 49: A Schematic of the assembled pipe and the pouring tank in Process II

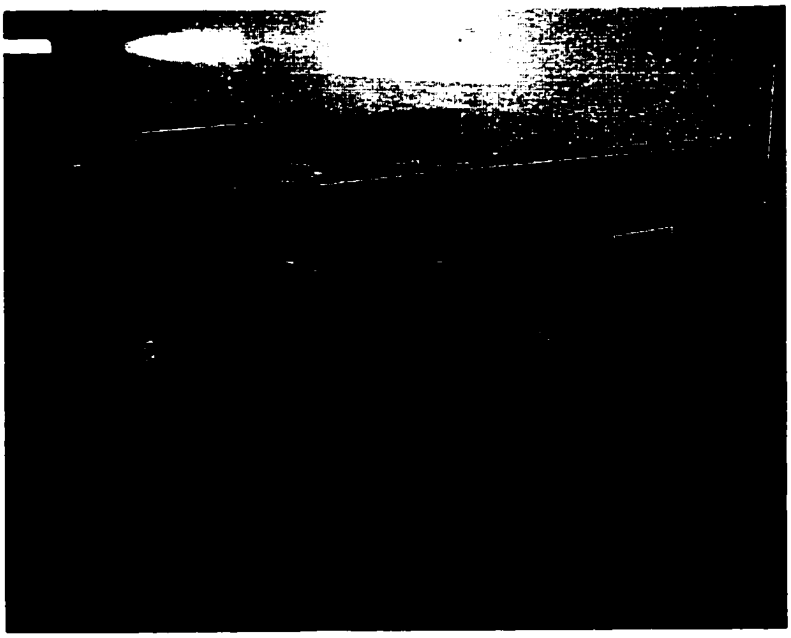


Figure 50: Photo illustrating the whole manufactured steel frame

Figure 51: Photo illustrating the wheel used to support and rotate the pipe

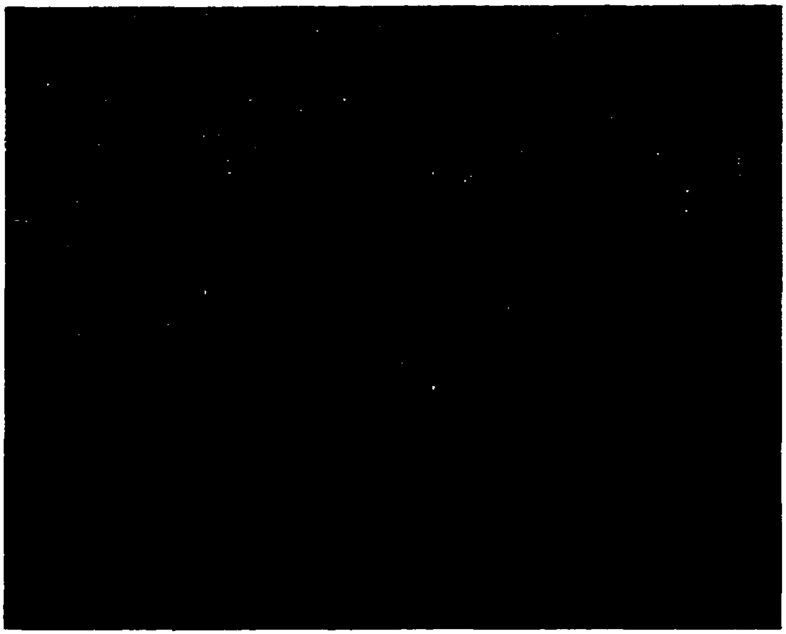


Figure 52: Photo illustrating the motor used to rotate the pipe

Figure 53: Photo illustrating the tire used to rotate the pipe through the motor

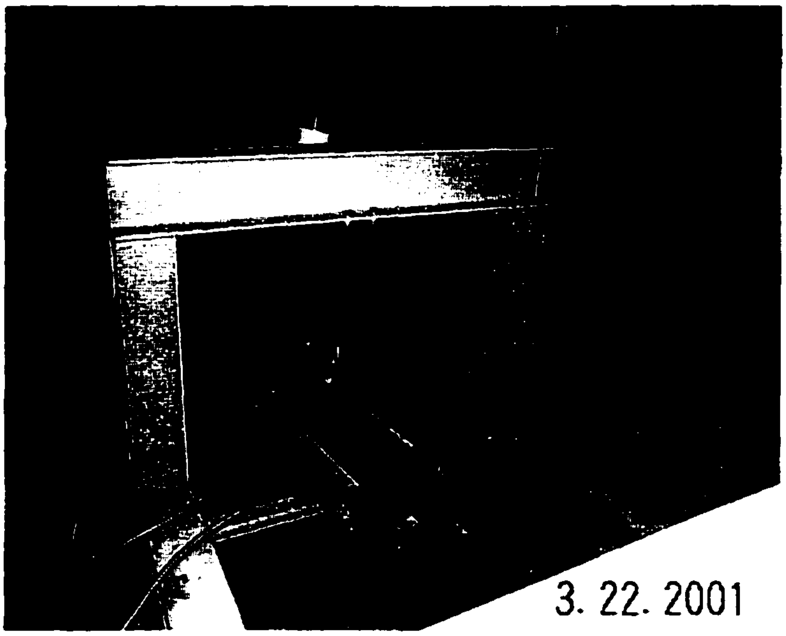

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Figure 54: Photo illustrating heating and pouring tank

Figure 55: Photo illustrating the pipe inside the frame

Figure 56: Photo illustrating a lined pipe (bell side) using the roll cast (Process II)

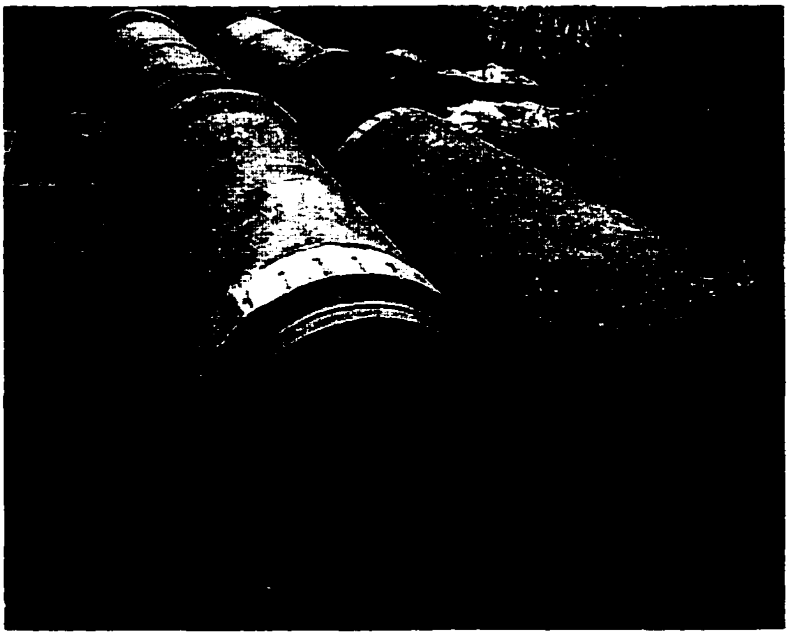

Figure 57: Photo illustrating a lined pipe (spigot side) using the roll cast (Process II)

Figure 58: Photo illustrating a cut section in a lined pipe using the roll cast (Process II)

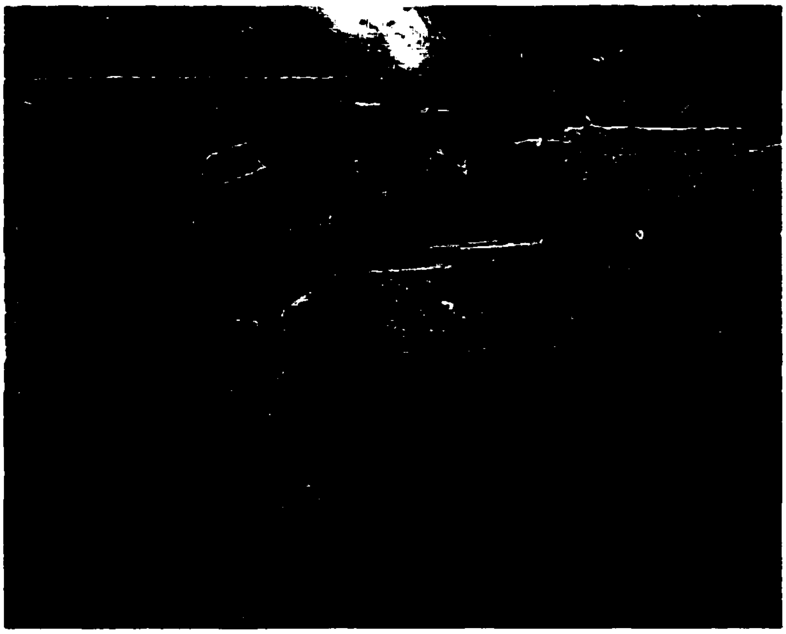

Figure 59: Photo illustrating a cut section in a lined pipe using the roll cast (Process II)

CONCLUSIONS

In general, the final result of this research was obtaining a sulfuric acid resistant pipe. Unmodified sulfur mortar was used to line the pipe and produce the new joints. In addition, during the laboratory study and the lining processes the following conclusions can be made.

- Modified sulfur mortar and sulfur mortar materials gave similar or even at some instances better compressive strength and provided superior sulfuric acid resistance than that of Portland cement concrete.
- The pouring temperature had little or no effect on bond strength of the material under ambient humidity conditions as indicated by both testing results and statistical analyses. The bond strength obtained using modified and unmodified sulfur mortars was adequate to provide proper bonding during the lining process.
- Polymer modified sulfur mortars are less susceptible to cracking due to shrinkage that will occur during cooling of regular sulfur. Laboratory test results show that modified sulfur mortars experienced less shrinkage than that of sulfur mortars. The following benefits are derived by using Dicyclopentadiene (DCP), using DCP gave best engineering properties, less sulfur odor, more ductility of the sulfur mortar (increased modulus). However, understanding the mechanical properties and controlling the reaction environment is an essential part of using DCP. In addition, the use of fiberglass in modified and unmodified sulfur mortars resulted in less shrinkage.
- The optimum DCP content from results of testing performed was 3% by weight of the sulfur. 5% modification did not result in significant changes in mechanical properties

beside the fact that the viscosity obtained affects obtaining a workable material.

- DCP modified sulfur is resistant to sulfuric acid. Fiberglass reinforcement is a beneficial additive for sulfur mortars, since it eliminates loss of compressive strength with time that occurred with all materials. However, statistical analysis did not provide enough evidence that the addition of fiberglass is beneficial to bond strength.
- This research shows the technical feasibility of using a sulfur mortar to provide an abrasion resistant barrier against exposure to sulfuric acid and the resulting deterioration.
- The aggregate in the base liner is mainly silica fines (sump that is considered waste material) and this material is not reactive with sulfuric acid and with minimal cost. Using silica-based aggregate is better than using carbonaceous aggregate that is reactive with sulfuric acid. Carbonaceous aggregates use is not recommended with environments were there is exposure to sulfuric acid.
- The proposed system has the potential to be applicable to more ranges of sizes and shapes in the reinforced concrete pipe industry, however, this needs further laboratory evaluation.
- Visual inspection of the pipe indicated that the sulfur mortar provides a smooth surface, loss of the cross-section of the pipe due to the presence of the lining is minimal and is compensated by the decrease in the surface roughness, and this will result in similar to larger discharge. In addition, less pore space than that observed in concrete was observed in the produced lining, which indicated less permeability than that of concrete, which has the possibility of reducing ground water contamination

due to sewer pipe leakage.

- Flexible jointing systems has been used while performing this research; this gives more versatility and acceptability to proposed system than the fixed jointing system.
- Use of sulfur liner has cost less than that of any other available alternatives. Based on price comparisons, other sulfuric acid resistant alternatives have shown to cost 30 to 100% more than sulfur mortar lined pipes.
- The fully lined pipe was manufactured by the roll cast mechanism and was lined with non-modified sulfur mortar, the quenching process provided a liner that did not crack and resulted in a fully lined pipe.

RECOMMENDATIONS

- Not all the working parameters have been completely developed; more experimentation is required in order to find the most efficient method of fabricating the lined pipe.
- Leakage prevention of the sulfur mortar from the mold system is a crucial step that needs to be taken care of before the lining process. Damaging the pipe is possible if this point is neglected.
- Accurate controls and mixing during the modification process are required. These parameters can be well maintained in small sized projects, but they are difficult to maintain on large scaled projects. More studies would be required for large-scale production.

APPENDIX A: LABORATORY TESTING RESULTS

Table A-1: Strength-time characteristics for pure Sulfur and aggregate

Time (hours)	Sample 1 Load (lb.)	Sample 2 Load (lb.)	Sample 3 Load (lb.)	Sample 1 Strength (psi)	Sample 2 Strength (psi)	Sample 3 Strength (psi)	Average Strength (psi)	Standard Deviation (psi)
Material: Pure Sulfur								
1.0	9400.0	10300.0	9900.0	2350.0	2575.0	2475.0	2466.7	112.7
24.0	12500.0	11200.0	11000.0	3125.0	2800.0	2750.0	2891.7	203.6
72.0	9900.0	10300.0	10700.0	2475.0	2575.0	2675.0	2575.0	100.0
168.0	7300.0	9200.0	7000.0	1825.0	2300.0	1750.0	1958.3	298.3
336.0	4900.0	7500.0	7400.0	1225.0	1875.0	1850.0	1650.0	368.3
Material: Pure Sulfur +10% Fine Aggregate								
1.0	9200.0	7400.0	7600.0	2300.0	1850.0	1900.0	2016.7	246.6
24.0	14800.0	16100.0	16500.0	3700.0	4025.0	4125.0	3950.0	222.2
72.0	13400.0	15000.0	16200.0	3350.0	3750.0	4050.0	3716.7	351.2
168.0	11800.0	10000.0	10700.0	2950.0	2500.0	2675.0	2708.3	226.8
336.0	4000.0	7500.0	7500.0	1000.0	1875.0	1875.0	1583.3	505.2
Material: Pure Sulfur +20% Fine Aggregate								
1.0	15000.0	15000.0	13700.0	3750.0	3750.0	3425.0	3641.7	187.6
24.0	17400.0	20200.0	19000.0	4350.0	5050.0	4750.0	4716.7	351.2
72.0	18500.0	17800.0	17300.0	4625.0	4450.0	4325.0	4466.7	150.7
168.0	20400.0	18000.0	16500.0	5100.0	4500.0	4125.0	4575.0	491.8
336.0	18500.0	19600.0	18100.0	4625.0	4900.0	4525.0	4683.3	194.2

Table A-1 (Continued): Strength-time characteristics for pure Sulfur and aggregate

Time (hours)	Sample 1 Load (lb.)	Sample 2 Load (lb.)	Sample 3 Load (lb.)	Sample 1 Strength (psi)	Sample 2 Strength (psi)	Sample 3 Strength (psi)	Average Strength (psi)	Standard Deviation (psi)
Material: Pure Sulfur +30% Fine Aggregate								
1.0	12500.0	12800.0	12700.0	3125.0	3200.0	3175.0	3166.7	38.2
24.0	17700.0	17200.0	17200.0	4425.0	4300.0	4300.0	4341.7	72.2
72.0	14400.0	13500.0	18500.0	3600.0	3375.0	4625.0	3866.7	666.3
168.0	13500.0	14000.0	15900.0	3375.0	3500.0	3975.0	3616.7	316.6
336.0	16300.0	17000.0	16000.0	4075.0	4250.0	4000.0	4108.3	128.3
Material: Pure Sulfur +40% Fine Aggregate								
1.0	21000.0	19400.0	20100.0	5250.0	4850.0	5025.0	5041.7	200.5
24.0	28000.0	28000.0	32000.0	7000.0	7000.0	8000.0	7333.3	577.4
72.0	25700.0	31200.0	28400.0	6425.0	7800.0	7100.0	7108.3	687.5
168.0	26000.0	27600.0	24500.0	6500.0	6900.0	6125.0	6508.3	387.6
336.0	30000.0	23200.0	21000.0	7500.0	5800.0	5250.0	6183.3	1173.0
Material: Pure Sulfur +40% Fine Aggregate +10% Coarse Aggregate								
1.0	28100.0	25800.0	25600.0	7025.0	6450.0	6400.0	6625.0	347.3
24.0	29800.0	30800.0	29000.0	7450.0	7700.0	7250.0	7466.7	225.5
72.0	32500.0	31700.0	31600.0	8125.0	7925.0	7900.0	7983.3	123.3
168.0	27100.0	22000.0	29600.0	6775.0	5500.0	7400.0	6558.3	968.4
336.0	28500.0	24500.0	24000.0	7125.0	6125.0	6000.0	6416.7	616.6

Table A-2: Strength-time characteristics for 1% Fiberglass reinforced pure Sulfur and aggregate

Time (hours)	Sample 1 Load (lb.)	Sample 2 Load (lb.)	Sample 3 Load (lb.)	Sample 1 Strength (psi)	Sample 2 Strength (psi)	Sample 3 Strength (psi)	Average Strength (psi)	Standard Deviation (psi)
Material: Pure Sulfur +1% Fiberglass								
1.0	21500.0	23100.0	21700.0	5375.0	5775.0	5425.0	5525.0	217.9
24.0	20000.0	19000.0	19200.0	5000.0	4750.0	4800.0	4850.0	132.3
72.0	21900.0	18500.0	17000.0	5475.0	4625.0	4250.0	4783.3	627.7
168.0	22200.0	20200.0	18700.0	5550.0	5050.0	4675.0	5091.7	439.0
336.0	18500.0	18500.0	20700.0	4625.0	4625.0	5175.0	4808.3	317.5
Material: Pure Sulfur +10% Fine Aggregate +1% Fiberglass								
1.0	14500.0	13500.0	14100.0	3625.0	3375.0	3525.0	3508.3	125.8
24.0	15700.0	17100.0	13500.0	3925.0	4275.0	3375.0	3858.3	453.7
72.0	15000.0	16000.0	15700.0	3750.0	4000.0	3925.0	3891.7	128.3
168.0	14200.0	16500.0	13000.0	3550.0	4125.0	3250.0	3641.7	444.6
336.0	16300.0	11200.0	10800.0	4075.0	2800.0	2700.0	3191.7	766.6
Material: Pure Sulfur +20% Fine Aggregate +1% Fiberglass								
1.0	18000.0	17000.0	18700.0	4500.0	4250.0	4675.0	4475.0	213.6
24.0	17800.0	18600.0	17500.0	4450.0	4650.0	4375.0	4491.7	142.2
72.0	13500.0	19000.0	17500.0	3375.0	4750.0	4375.0	4166.7	710.8
168.0	16000.0	17500.0	12000.0	4000.0	4375.0	3000.0	3791.7	710.8
336.0	20400.0	20000.0	21100.0	5100.0	5000.0	5275.0	5125.0	139.2

Table A-2 (Continued): Strength-Time characteristics for 1% Fiberglass reinforced pure Sulfur and aggregate

Time (hours)	Sample 1 Load (lb.)	Sample 2 Load (lb.)	Sample 3 Load (lb.)	Sample 1 Strength (psi)	Sample 2 Strength (psi)	Sample 3 Strength (psi)	Average Strength (psi)	Standard Deviation (psi)
Material: Pure Sulfur +30% Fine Aggregate +1% Fiberglass								
1.0	18000.0	17000.0	18700.0	4500.0	4250.0	4675.0	4475.0	213.6
24.0	17800.0	18600.0	17500.0	4450.0	4650.0	4375.0	4491.7	142.2
72.0	13500.0	19000.0	17500.0	3375.0	4750.0	4375.0	4166.7	710.8
168.0	16000.0	17500.0	12000.0	4000.0	4375.0	3000.0	3791.7	710.8
336.0	20400.0	20000.0	21100.0	5100.0	5000.0	5275.0	5125.0	139.2
Material: Pure Sulfur +40% Fine Aggregate +1% Fiberglass								
1.0	21500.0	23100.0	22600.0	5375.0	5775.0	5650.0	5600.0	204.6
24.0	24000.0	28200.0	24200.0	6000.0	7050.0	6050.0	6366.7	592.3
72.0	26200.0	25500.0	25500.0	6550.0	6375.0	6375.0	6433.3	101.0
168.0	26100.0	22500.0	25700.0	6525.0	5625.0	6425.0	6191.7	493.3
336.0	26200.0	27400.0	28200.0	6550.0	6850.0	7050.0	6816.7	251.7
Material: Pure Sulfur +40% Fine Aggregate +10% Coarse Aggregate +1% Fiberglass								
1.0	31400.0	31100.0	28900.0	7850.0	7775.0	7225.0	7616.7	341.3
24.0	33500.0	33500.0	33700.0	8375.0	8375.0	8425.0	8391.7	28.9
72.0	34700.0	32200.0	30400.0	8675.0	8050.0	7600.0	8108.3	539.9
168.0	32200.0	29900.0	28500.0	8050.0	7475.0	7125.0	7550.0	467.0
336.0	30200.0	31700.0	26700.0	7550.0	7925.0	6675.0	7383.3	641.5

Table A-3: Strength-time characteristics for 3% Dicyclopentadiene (DCP) modified Sulfur and aggregate

Time (hours)	Sample 1 Load (lb.)	Sample 2 Load (lb.)	Sample 3 Load (lb.)	Sample 1 Strength (psi)	Sample 2 Strength (psi)	Sample 3 Strength (psi)	Average Strength (psi)	Standard Deviation (psi.)
				Material: Sulfur +3% DCP				
1.0	6000.0	6500.0	6000.0	1500.0	1625.0	1500.0	1541.7	72.2
24.0	14600.0	15000.0	15000.0	3650.0	3750.0	3750.0	3716.7	57.7
72.0	16100.0	17200.0	17200.0	4025.0	4300.0	4300.0	4208.3	158.8
168.0	15200.0	16700.0	16000.0	3800.0	4175.0	4000.0	3991.7	187.6
336.0	16700.0	16900.0	15000.0	4175.0	4225.0	3750.0	4050.0	261.0
				Material: Pure Sulfur +3% DCP +10% Fine Aggregate				
1.0	11300.0	10000.0	9600.0	2825.0	2500.0	2400.0	2575.0	222.2
24.0	16700.0	18500.0	19700.0	4175.0	4625.0	4925.0	4575.0	377.5
72.0	18100.0	17800.0	17800.0	4525.0	4450.0	4450.0	4475.0	43.3
168.0	17700.0	17700.0	18300.0	4425.0	4425.0	4575.0	4475.0	86.6
336.0	18700.0	19200.0	18700.0	4675.0	4800.0	4675.0	4716.7	72.2
				Material: Pure Sulfur +3% DCP +20% Fine Aggregate				
1.0	21300.0	22600.0	22500.0	5325.0	5650.0	5625.0	5533.3	180.9
24.0	27300.0	27600.0	27100.0	6825.0	6900.0	6775.0	6833.3	62.9
72.0	26500.0	25400.0	25500.0	6625.0	6350.0	6375.0	6450.0	152.1
168.0	22000.0	22200.0	22800.0	5500.0	5550.0	5700.0	5583.3	104.1
336.0	23200.0	25000.0	23700.0	5800.0	6250.0	5925.0	5991.7	232.3

Table A-3 (Continued): Strength-time characteristics for 3% Dicyclopentadiene (DCP) modified Sulfur and aggregate

Time (hours)	Sample 1 Load (lb.)	Sample 2 Load (lb.)	Sample 3 Load (lb.)	Sample 1 Strength (psi)	Sample 2 Strength (psi)	Sample 3 Strength (psi)	Average Strength (psi)	Standard Deviation (psi)
			Material: Pure Sulfur +3% DCP +30% Fine Aggregate					
1.0	29000.0	28700.0	30000.0	7250.0	7175.0	7500.0	7308.3	170.2
24.0	27300.0	27600.0	27100.0	6825.0	6900.0	6775.0	6833.3	62.9
72.0	26500.0	25400.0	25500.0	6625.0	6350.0	6375.0	6450.0	152.1
168.0	27500.0	29600.0	27700.0	6875.0	7400.0	6925.0	7066.7	289.8
336.0	28700.0	29700.0	28200.0	7175.0	7425.0	7050.0	7216.7	190.9
			Material: Pure Sulfur +3% DCP +40% Fine Aggregate					
1.0	29000.0	28700.0	30000.0	7250.0	7175.0	7500.0	7308.3	170.2
24.0	33000.0	31700.0	33000.0	8250.0	7925.0	8250.0	8141.7	187.6
72.0	32600.0	31300.0	32800.0	8150.0	7825.0	8200.0	8058.3	203.6
168.0	30800.0	32100.0	32500.0	7700.0	8025.0	8125.0	7950.0	222.2
336.0	33200.0	29800.0	31800.0	8300.0	7450.0	7950.0	7900.0	427.2

Table A-4: Strength-time characteristics for 1% Fiberglass reinforced 3% DCP modified Sulfur and aggregate

Time (hours)	Sample 1 Load (lb.)	Sample 2 Load (lb.)	Sample 3 Load (lb.)	Sample 1 Strength (psi)	Sample 2 Strength (psi)	Sample 3 Strength (psi)	Average Strength (psi)	Standard Deviation (psi)
Material: Pure Sulfur +3% DCP +1% Fiberglass								
1.0	4500.0	4200.0	4400.0	1125.0	1050.0	1100.0	1091.7	38.2
24.0	8500.0	9000.0	8800.0	2125.0	2250.0	2200.0	2191.7	62.9
72.0	9600.0	10000.0	9800.0	2400.0	2500.0	2450.0	2450.0	50.0
168.0	11000.0	12400.0	11700.0	2750.0	3100.0	2925.0	2925.0	175.0
336.0	12000.0	12000.0	11900.0	3000.0	3000.0	2975.0	2991.7	14.4
Material: Pure Sulfur +3% DCP +10% Fine Aggregate +1% Fiberglass								
1.0	6000.0	6000.0	5900.0	1500.0	1500.0	1475.0	1491.7	14.4
24.0	9500.0	9200.0	9300.0	2375.0	2300.0	2325.0	2333.3	38.2
72.0	10600.0	11000.0	10600.0	2650.0	2750.0	2650.0	2683.3	57.7
168.0	13000.0	13200.0	13000.0	3250.0	3300.0	3250.0	3266.7	28.9
336.0	14800.0	13700.0	14000.0	3700.0	3425.0	3500.0	3541.7	142.2
Material: Pure Sulfur +3% DCP +20% Fine Aggregate +1% Fiberglass								
1.0	9700.0	10800.0	10300.0	2425.0	2700.0	2575.0	2566.7	137.7
24.0	20500.0	22200.0	21300.0	5125.0	5550.0	5325.0	5333.3	212.6
72.0	23100.0	24600.0	24000.0	5775.0	6150.0	6000.0	5975.0	188.7
168.0	26500.0	26200.0	26200.0	6625.0	6550.0	6550.0	6575.0	43.3
336.0	29200.0	29200.0	29100.0	7300.0	7300.0	7275.0	7291.7	14.4

Table A-4 (Continued): Strength-time characteristics for 1% Fiberglass reinforced 3% DCP modified Sulfur and aggregate

Time (hours)	Sample 1 Load (lb.)	Sample 2 Load (lb.)	Sample 3 Load (lb.)	Sample 1 Strength (psi)	Sample 2 Strength (psi)	Sample 3 Strength (psi)	Average Strength (psi)	Standard Deviation (psi)
Material: Pure Sulfur +3% DCP +30% Fine Aggregate +1% Fiberglass								
1.0	13800.0	14700.0	14200.0	3450.0	3675.0	3550.0	3558.3	112.7
24.0	20500.0	24300.0	21300.0	5125.0	6075.0	5325.0	5508.3	500.8
72.0	21300.0	22600.0	22600.0	5325.0	5650.0	5650.0	5541.7	187.6
168.0	25800.0	24400.0	24400.0	6450.0	6100.0	6100.0	6216.7	202.1
336.0	23700.0	22500.0	22900.0	5925.0	5625.0	5725.0	5758.3	152.8
Material: Pure Sulfur +3% DCP +40% Fine Aggregate +1% Fiberglass								
1.0	21000.0	22000.0	21600.0	5250.0	5500.0	5400.0	5383.3	125.8
24.0	31600.0	33000.0	32800.0	7900.0	8250.0	8200.0	8116.7	189.3
72.0	32200.0	25400.0	33200.0	8050.0	6350.0	8300.0	7566.7	1061.1
168.0	32000.0	35000.0	24000.0	8000.0	8750.0	6000.0	7583.3	1421.6
336.0	38000.0	38200.0	32000.0	9500.0	9550.0	8000.0	9016.7	880.8
Material: Pure Sulfur +3% DCP +40% Fine Aggregate +10% Coarse Aggregate +1% Fiberglass								
1.0	21200.0	22300.0	18900.0	5300.0	5575.0	4725.0	5200.0	433.7
24.0	19800.0	25200.0	26100.0	4950.0	6300.0	6525.0	5925.0	851.8
72.0	32400.0	33600.0	34200.0	8100.0	8400.0	8550.0	8350.0	229.1
168.0	34200.0	34300.0	28800.0	8550.0	8575.0	7200.0	8108.3	786.7
336.0	37000.0	37800.0	33600.0	9250.0	9450.0	8400.0	9033.3	557.5

Table A-5: Strength-time characteristics for 5% Dicyclopentadiene (DCP) modified Sulfur and aggregate

Time (hours)	Sample 1 Load (lb.)	Sample 2 Load (lb.)	Sample 3 Load (lb.)	Sample 1 Strength (psi)	Sample 2 Strength (psi)	Sample 3 Strength (psi)	Average Strength (psi)	Standard Deviation (psi)
Material: Sulfur +5% DCP								
1.0	2000.0	1500.0	1700.0	500.0	375.0	425.0	433.3	62.9
24.0	2700.0	2800.0	2800.0	675.0	700.0	700.0	691.7	14.4
72.0	4600.0	4400.0	4600.0	1150.0	1100.0	1150.0	1133.3	28.9
168.0	5200.0	5000.0	5000.0	1300.0	1250.0	1250.0	1266.7	28.9
336.0	4500.0	4600.0	4600.0	1125.0	1150.0	1150.0	1141.7	14.4
Material: Pure Sulfur +5% DCP +10% Fine Aggregate								
1.0	2500.0	3000.0	2700.0	625.0	750.0	675.0	683.3	62.9
24.0	2700.0	2800.0	2800.0	675.0	700.0	700.0	691.7	14.4
72.0	6500.0	6600.0	6500.0	1625.0	1650.0	1625.0	1633.3	14.4
168.0	6700.0	7100.0	6800.0	1675.0	1775.0	1700.0	1716.7	52.0
336.0	5400.0	5900.0	5800.0	1350.0	1475.0	1450.0	1425.0	66.1
Material: Pure Sulfur +5% DCP +20% Fine Aggregate								
1.0	5000.0	4600.0	4800.0	1250.0	1150.0	1200.0	1200.0	50.0
24.0	6200.0	6100.0	6100.0	1550.0	1525.0	1525.0	1533.3	14.4
72.0	7800.0	8300.0	8000.0	1950.0	2075.0	2000.0	2008.3	62.9
168.0	9700.0	9200.0	9500.0	2425.0	2300.0	2375.0	2366.7	62.9
336.0	6400.0	7300.0	7000.0	1600.0	1825.0	1750.0	1725.0	114.6

Table A-5 (Continued): Strength-time characteristics for 5% Dicyclopentadiene (DCP) modified Sulfur and aggregate

Time (hours)	Sample 1 Load (lb.)	Sample 2 Load (lb.)	Sample 3 Load (lb.)	Sample 1 Strength (psi)	Sample 2 Strength (psi)	Sample 3 Strength (psi)	Average Strength (psi)	Standard Deviation (psi)
Material: Pure Sulfur +5% DCP +30% Fine Aggregate								
1.0	6000.0	7600.0	6800.0	1500.0	1900.0	1700.0	1700.0	200.0
24.0	9800.0	9800.0	9900.0	2450.0	2450.0	2475.0	2458.3	14.4
72.0	10300.0	9800.0	9900.0	2575.0	2450.0	2475.0	2500.0	66.1
168.0	9700.0	10900.0	10000.0	2425.0	2725.0	2500.0	2550.0	156.1
336.0	8800.0	10200.0	10100.0	2200.0	2550.0	2525.0	2425.0	195.3
Material: Pure Sulfur +5% DCP +40% Fine Aggregate								
1.0	8800.0	7700.0	8300.0	2200.0	1925.0	2075.0	2066.7	137.7
24.0	13300.0	12800.0	13300.0	3325.0	3200.0	3325.0	3283.3	72.2
72.0	15200.0	13800.0	14100.0	3800.0	3450.0	3525.0	3591.7	184.3
168.0	16500.0	15700.0	16000.0	4125.0	3925.0	4000.0	4016.7	101.0
336.0	13600.0	13500.0	13500.0	3400.0	3375.0	3375.0	3383.3	14.4
Material: Pure Sulfur +5% DCP +40% Fine Aggregate +10% Coarse Aggregate								
1.0	12000.0	12400.0	12200.0	3000.0	3100.0	3050.0	3050.0	50.0
24.0	15000.0	15000.0	14700.0	3750.0	3750.0	3675.0	3725.0	43.3
72.0	17100.0	18200.0	18500.0	4275.0	4550.0	4625.0	4483.3	184.3
168.0	19500.0	18600.0	19000.0	4875.0	4650.0	4750.0	4758.3	112.7
336.0	19700.0	19200.0	20600.0	4925.0	4800.0	5150.0	4958.3	177.4

Table A-6: Strength-time characteristics for 1% Fiberglass reinforced 5% DCP modified Sulfur and aggregate

Time (hours)	Sample 1 Load (lb.)	Sample 2 Load (lb.)	Sample 3 Load (lb.)	Sample 1 Strength (psi)	Sample 2 Strength (psi)	Sample 3 Strength (psi)	Average Strength (psi)	Standard Deviation (psi)
Material: Pure Sulfur +5% DCP +1% Fiberglass								
1.0	6500.0	7400.0	5900.0	1625.0	1850.0	1475.0	1650.0	188.7
24.0	14000.0	14600.0	13900.0	3500.0	3650.0	3475.0	3541.7	94.6
72.0	15600.0	15400.0	15300.0	3900.0	3850.0	3825.0	3858.3	38.2
168.0	18200.0	17000.0	17200.0	4550.0	4250.0	4300.0	4366.7	160.7
336.0	14900.0	15000.0	14500.0	3725.0	3750.0	3625.0	3700.0	66.1
Material: Pure Sulfur +5% DCP +10% Fine Aggregate +1% Fiberglass								
1.0	8200.0	8500.0	8300.0	2050.0	2125.0	2075.0	2083.3	38.2
24.0	22600.0	20200.0	21000.0	5650.0	5050.0	5250.0	5316.7	305.5
72.0	19200.0	20600.0	18700.0	4800.0	5150.0	4675.0	4875.0	246.2
168.0	22800.0	23000.0	21900.0	5700.0	5750.0	5475.0	5641.7	146.5
336.0	18700.0	21400.0	21500.0	4675.0	5350.0	5375.0	5133.3	397.1
Material: Pure Sulfur +5% DCP +20% Fine Aggregate +1% Fiberglass								
1.0	9000.0	10200.0	9500.0	2250.0	2550.0	2375.0	2391.7	150.7
24.0	23000.0	24400.0	23600.0	5750.0	6100.0	5900.0	5916.7	175.6
72.0	23200.0	24400.0	23700.0	5800.0	6100.0	5925.0	5941.7	150.7
168.0	22600.0	22800.0	22600.0	5650.0	5700.0	5650.0	5666.7	28.9
336.0	26600.0	24800.0	24900.0	6650.0	6200.0	6225.0	6358.3	252.9

Table A-6 (Continued): Strength-time characteristics for 1% Fiberglass reinforced 5% DCP modified Sulfur and aggregate

Time (hours)	Sample 1 Load (lb.)	Sample 2 Load (lb.)	Sample 3 Load (lb.)	Sample 1 Strength (psi)	Sample 2 Strength (psi)	Sample 3 Strength (psi)	Average Strength (psi)	Standard Deviation (psi)
Material: Pure Sulfur +5% DCP +30% Fine Aggregate +1% Fiberglass								
1.0	11000.0	13000.0	11500.0	2750.0	3250.0	2875.0	2958.3	260.2
24.0	26200.0	28200.0	26500.0	6550.0	7050.0	6625.0	6741.7	269.6
72.0	27200.0	26400.0	26800.0	6800.0	6600.0	6700.0	6700.0	100.0
168.0	30000.0	29800.0	29500.0	7500.0	7450.0	7375.0	7441.7	62.9
336.0	31100.0	30800.0	30600.0	7775.0	7700.0	7650.0	7708.3	62.9
Material: Pure Sulfur +5% DCP +40% Fine Aggregate +1% Fiberglass								
1.0	13000.0	12500.0	12700.0	3250.0	3125.0	3175.0	3183.3	62.9
24.0	30400.0	30200.0	30200.0	7600.0	7550.0	7550.0	7566.7	28.9
72.0	30500.0	29700.0	30000.0	7625.0	7425.0	7500.0	7516.7	101.0
168.0	30100.0	29800.0	29900.0	7525.0	7450.0	7475.0	7483.3	38.2
336.0	32800.0	33000.0	33000.0	8200.0	8250.0	8250.0	8233.3	28.9
Material: Pure Sulfur +5% DCP +40% Fine Aggregate +10% Coarse Aggregate +1% Fiberglass								
1.0	17100.0	18400.0	17700.0	4275.0	4600.0	4425.0	4433.3	162.7
24.0	31000.0	30500.0	30700.0	7750.0	7625.0	7675.0	7683.3	62.9
72.0	29300.0	31700.0	30400.0	7325.0	7925.0	7600.0	7616.7	300.3
168.0	33800.0	32400.0	33000.0	8450.0	8100.0	8250.0	8266.7	175.6
336.0	34500.0	32200.0	33100.0	8625.0	8050.0	8275.0	8316.7	289.8

Table A-7: Shrinkage results for pure Sulfur

Time (minutes)	Shrinkage Strain, Sample 1, %	Shrinkage Strain, Sample 2, %	Shrinkage Strain, Average, %
0	0.0000	0.0000	0.0000
0.25	0.2000	0.1800	0.1900
0.50	0.2200	0.2000	0.2100
0.67	0.2850	0.2740	0.2795
0.83	0.3400	0.3270	0.3335
1.00	0.3700	0.3610	0.3655
1.50	0.3990	0.3990	0.3990
2.00	0.4050	0.4010	0.4030
2.50	0.4050	0.4010	0.4030
3.00	0.4050	0.4030	0.4040
4.00	0.4050	0.4030	0.4040
8.00	0.4430	0.4460	0.4445
24	0.4970	0.4860	0.4915
48	0.5260	0.5100	0.5180
72	0.5440	0.5330	0.5385
96	0.5470	0.5340	0.5405
120	0.5470	0.5350	0.5410
144	0.5480	0.5360	0.5420

Table A-8: Shrinkage results for pure Sulfur + 40%, fine aggregate +10% coarse aggregate

Time (minutes)	Shrinkage Strain, Sample 1, %	Shrinkage Strain, Sample 2, %	Shrinkage Strain, Average, %
0	0.0000	0.0000	0.0000
0.25	0.1800	0.1770	0.1785
0.50	0.2130	0.2075	0.2103
0.67	0.2260	0.2200	0.2230
0.83	0.2350	0.2290	0.2320
1.00	0.2390	0.2325	0.2358
1.50	0.2460	0.2395	0.2428
2.00	0.2470	0.2425	0.2448
2.50	0.2480	0.2435	0.2458
3.00	0.2500	0.2460	0.2480
4.00	0.2560	0.2490	0.2525
8.00	0.2730	0.2685	0.2708
24	0.2910	0.2865	0.2888
48	0.2960	0.2890	0.2925
72	0.3000	0.2955	0.2978
96	0.3000	0.2955	0.2978
120	0.3000	0.2955	0.2978
144	0.3000	0.2955	0.2978

Table A-9: Shrinkage results for pure Sulfur + 40%, fine aggregate + 10% coarse aggregate + 1% Fiberglass reinforcement

Time (minutes)	Shrinkage Strain, Sample 1, %	Shrinkage Strain, Sample 2, %	Shrinkage Strain, Average, %
0	0.0000	0.0000	0.0000
0.25	0.1700	0.1730	0.1715
0.50	0.2170	0.2100	0.2135
0.67	0.2320	0.2250	0.2285
0.83	0.2430	0.2280	0.2355
1.00	0.2480	0.2330	0.2405
1.50	0.2530	0.2380	0.2455
2.00	0.2590	0.2420	0.2505
2.50	0.2610	0.2420	0.2515
3.00	0.2610	0.2420	0.2515
4.00	0.2680	0.2420	0.2550
8.00	0.2760	0.2460	0.2610
24	0.2810	0.2550	0.2680
48	0.2810	0.2580	0.2695
72	0.2830	0.2580	0.2705
96	0.2920	0.2600	0.2760
120	0.2920	0.2600	0.2760
144	0.2920	0.2600	0.2760

Table A-10: Shrinkage results for 3% DCP modified Sulfur

Time (minutes)	Shrinkage Strain, Sample 1, %	Shrinkage Strain, Sample 2, %	Shrinkage Strain, Average, %
0	0.0000	0.0000	0.0000
0.50	0.1800	0.1490	0.1645
0.67	0.2070	0.1740	0.1905
0.83	0.2270	0.1940	0.2105
1.00	0.2360	0.2050	0.2205
1.50	0.2460	0.2150	0.2305
2.00	0.2470	0.2190	0.2330
2.50	0.2480	0.2190	0.2335
3.00	0.2490	0.2210	0.2350
4.00	0.2580	0.2270	0.2425
8.00	0.2400	0.2500	0.2450
24	0.3640	0.3390	0.3515
48	0.3640	0.3990	0.3815
72	0.3630	0.3990	0.3810
96	0.3620	0.3970	0.3795

Table A-11: Shrinkage results for 3% DCP modified Sulfur + 40%, fine aggregate + 10% coarse aggregate

Time (minutes)	Shrinkage Strain, Sample 1, %	Shrinkage Strain, Sample 2, %	Shrinkage Strain, Average, %
0	0.0000	0.0000	0.0000
0.50	0.1500	0.1470	0.1485
0.67	0.1690	0.2250	0.1970
0.83	0.1780	0.2420	0.2100
1.00	0.1860	0.2540	0.2200
1.50	0.1880	0.2650	0.2265
2.00	0.1900	0.2670	0.2285
2.50	0.1820	0.2680	0.2250
3.00	0.1920	0.2710	0.2315
4.00	0.1930	0.2710	0.2320
8.00	0.2000	0.2720	0.2360
24	0.2130	0.2790	0.2460
48	0.2190	0.2900	0.2545
72	0.2190	0.2970	0.2580
96	0.2150	0.2940	0.2545

Table A-12: Shrinkage results for 3% DCP modified Sulfur + 40%, fine aggregate + 10% coarse aggregate + 1% Fiberglass reinforcement

Time (minutes)	Shrinkage Strain, Sample 1, %	Shrinkage Strain, Sample 2, %	Shrinkage Strain, Average, %
0	0.0000	0.0000	0.0000
0.50	0.1500	0.1470	0.1485
0.67	0.1760	0.1650	0.1705
0.83	0.1890	0.1760	0.1825
1.00	0.1960	0.1810	0.1885
1.50	0.2010	0.1840	0.1925
2.00	0.2020	0.1850	0.1935
2.50	0.2030	0.1860	0.1945
3.00	0.2030	0.1860	0.1945
4.00	0.2040	0.1860	0.1950
8.00	0.2060	0.1890	0.1975
24	0.1980	0.1820	0.1900
48	0.2010	0.1850	0.1930
72	0.1950	0.1810	0.1880
96	0.1920	0.1790	0.1855

Table A-13: Shrinkage results for 5% DCP modified Sulfur

Time (minutes)	Shrinkage Strain, Sample 1, %	Shrinkage Strain, Sample 2, %	Shrinkage Strain, Average, %
0	0.0000	0.0000	0.0000
0.25	0.0160	0.0160	0.0160
0.50	0.1290	0.1310	0.1300
0.67	0.1530	0.1540	0.1535
0.83	0.1720	0.1720	0.1720
1.00	0.1800	0.1810	0.1805
1.50	0.1890	0.1890	0.1890
2.00	0.1910	0.1930	0.1920
2.50	0.1930	0.1950	0.1940
3.00	0.1940	0.1960	0.1950
4.00	0.1950	0.1970	0.1960
8.00	0.2020	0.2050	0.2035
24	0.2200	0.2230	0.2215
48	0.2250	0.2280	0.2265
72	0.2290	0.2320	0.2305
96	0.2290	0.2320	0.2305
120	0.2290	0.2320	0.2305
144	0.2290	0.2320	0.2305

Table A-14: Shrinkage results for 5% DCP modified Sulfur + 40% fine aggregate + 10% coarse aggregate

Time (minutes)	Shrinkage Strain, Sample 1, %	Shrinkage Strain, Sample 2, %	Shrinkage Strain, Average, %
0	0.0000	0.0000	0.0000
0.25	0.0180	0.0160	0.0170
0.50	0.0250	0.0220	0.0235
0.67	0.0350	0.0380	0.0365
0.83	0.0640	0.0670	0.0655
1.00	0.0750	0.0790	0.0770
1.50	0.0880	0.0900	0.0890
2.00	0.0900	0.0920	0.0910
2.50	0.0930	0.0940	0.0935
3.00	0.0950	0.0940	0.0945
4.00	0.0970	0.0950	0.0960
8.00	0.0990	0.0980	0.0985
24	0.1100	0.1160	0.1130
48	0.1150	0.1210	0.1180
72	0.1200	0.1250	0.1225
96	0.1200	0.1250	0.1225
120	0.1300	0.1250	0.1275
144	0.1300	0.1250	0.1275

Table A-15: Shrinkage results for 5% DCP modified Sulfur + 40% fine aggregate + 10% coarse aggregate + 1% Fiberglass reinforcement

Time (minutes)	Shrinkage Strain, Sample 1, %	Shrinkage Strain, Sample 2, %	Shrinkage Strain, Average, %
0	0.0000	0.0000	0.0000
0.25	0.0160	0.0160	0.0160
0.50	0.0510	0.0440	0.0475
0.67	0.0620	0.0560	0.0590
0.83	0.0710	0.0650	0.0680
1.00	0.0770	0.0680	0.0725
1.50	0.0820	0.0750	0.0785
2.00	0.0830	0.0800	0.0815
2.50	0.0840	0.0810	0.0825
3.00	0.0860	0.0840	0.0850
4.00	0.0920	0.0840	0.0880
8.00	0.1080	0.1060	0.1070
24	0.1270	0.1240	0.1255
48	0.1320	0.1240	0.1280
72	0.1360	0.1330	0.1345
96	0.1360	0.1330	0.1345
120	0.1360	0.1330	0.1345
144	0.1360	0.1330	0.1345

Table A-16: Bond strength results for pure Sulfur (Pouring temperature 270 °F)

Bonding Material	Sample No.	Load (lb.)	Dimension 1, +	Dimension 2, +	Dimension -	Area (in^2)	Bond Strength (psi)	Average Strength (psi)	Standard Deviation (psi)
Sulfur	1	500.00	2.50	2.50	0.00	6.25	80.00		
	2	500.00	2.50	2.60	0.00	6.50	76.92		
	3	450.00	2.50	2.50	0.00	6.25	72.00	76.31	4.04
Sulfur +50% Aggregate	1	900.00	2.50	2.60	0.00	6.50	138.46		
	2	850.00	2.50	2.50	0.00	6.25	136.00		
	3	850.00	2.50	2.50	0.00	6.25	136.00	136.82	1.42
Sulfur +50% Aggregate + 1% Fiber Glass	1	800.00	2.50	2.50	0.00	6.25	128.00		
	2	800.00	2.60	2.50	0.00	6.50	123.08		
	3	900.00	2.60	2.60	0.00	6.76	133.14	128.07	5.03

Table A-17: Bond strength results for 3% DCP modified Sulfur (Pouring temperature 270 °F)

Bonding Material	Sample No.	Load (lb.)	Dimension 1, +	Dimension 2, +	Dimension -	Area (in^2)	Bond Strength (psi)	Average Strength (psi)	Standard Deviation (psi)
Sulfur +3% DCP	1	850.00	2.40	2.40	0.50	5.26	161.60		
	2	800.00	2.40	2.40	0.50	5.26	152.09		
	3	800.00	2.30	2.40	0.00	5.52	144.93	152.87	8.36
Sulfur +3% DCP + 50% Aggregate	1	1000.00	2.40	2.40	0.00	5.76	173.61		
	2	1025.00	2.40	2.50	0.00	6.00	170.83		
	3	1050.00	2.50	2.50	0.00	6.25	168.00	170.81	2.81
Sulfur +3% DCP +50% Aggregate +1% Fiber Glass	1	800.00	2.50	2.40	0.00	6.00	133.33		
	2	800.00	2.45	2.50	0.00	6.13	130.61		
	3	700.00	2.40	2.45	0.00	5.88	119.05	127.66	7.59

Table A-18: Bond strength results for 3% DCP, 1% Fiberglass reinforced modified Sulfur (Pouring temperature 270 °F)

Bonding Material	Sample No.	Load (lb.)	Dimension 1, +	Dimension 2, +	Dimension -	Area (in^2)	Bond Strength (psi)	Average Strength (psi)	Standard Deviation (psi)
Sulfur +5% DCP	1	800.00	2.40	2.40	0.00	5.76	138.89		
	2	700.00	2.40	2.50	0.00	6.00	116.67		
	3	700.00	2.50	2.40	0.00	6.00	116.67	124.07	12.83
Sulfur +5% DCP +50% Aggregate	1	850.00	2.40	2.50	0.00	6.00	141.67		
	2	1000.00	2.50	2.50	0.00	6.25	160.00		
	3	950.00	2.40	2.50	0.90	5.10	186.27	162.65	22.42
Sulfur +5% DCP +50% Aggregate +1% Fiber Glass	1	850.00	2.50	2.50	0.00	6.25	136.00		
	2	750.00	2.50	2.40	0.00	6.00	125.00		
	3	850.00	2.40	2.50	0.00	6.00	141.67	134.22	8.47

Table A-19: Bond strength results for pure Sulfur (Pouring temperature 290 °F)

Bonding Material	Sample No.	Load (lb.)	Dimension 1, +	Dimension 2, +	Dimension -	Area (in^2)	Bond Strength (psi)	Average Strength (psi)	Standard Deviation (psi)
Sulfur	1	800.00	2.80	2.60	0.00	7.28	109.89		
	2	850.00	2.70	2.80	0.00	7.56	112.43		
	3	950.00	2.60	2.60	0.00	6.76	140.53	120.95	17.00
Sulfur +50% Aggregate	1	1200.00	2.60	2.65	0.00	6.89	174.17		
	2	950.00	2.60	2.55	0.00	6.63	143.29		
	3	1200.00	2.65	2.70	0.00	7.16	167.71	161.72	16.29
Sulfur +50% Aggregate +1% Fiber Glass	1	700.00	2.50	2.60	0.00	6.50	107.69		
	2	800.00	2.50	2.60	0.00	6.50	123.08		
	3	1000.00	2.50	2.75	0.00	6.88	145.45	125.41	18.99

Table A-20: Bond strength results for 3% DCP modified Sulfur (Pouring temperature 290 °F)

Bonding Material	Sample No.	Load (lb.)	Dimension 1, +	Dimension 2, +	Dimension -	Area (in^2)	Bond Strength (psi)	Average Strength (psi)	Standard Deviation (psi)
Sulfur +3% DCP	1	800.00	2.50	2.50	0.00	6.25	128.00		
	2	800.00	2.50	2.50	0.00	6.25	128.00		
	3	800.00	2.50	2.50	0.00	6.25	128.00	128.00	0.00
Sulfur +3% DCP +50% Aggregate	1	1050.00	2.50	2.50	0.00	6.25	168.00		
	2	950.00	2.50	2.50	0.00	6.25	152.00		
	3	1000.00	2.50	2.50	0.00	6.25	160.00	160.00	8.00
Sulfur +3% DCP +50% Aggregate +1% Fiber Glass	1	900.00	2.50	2.50	0.00	6.25	144.00		
	2	1050.00	2.50	2.50	0.00	6.25	168.00		
	3	1100.00	2.50	2.50	0.00	6.25	176.00	162.67	16.65

Table A-21: Bond strength results for 3% DCP, 1% fiberglass reinforced modified Sulfur (Pouring temperature 290 °F)

Bonding Material	Sample No.	Load (lb.)	Dimension 1, +	Dimension 2, +	Dimension -	Area (in^2)	Bond Strength (psi)	Average Strength (psi)	Standard Deviation (psi)
Sulfur +5% DCP	1	800.00	2.50	2.50	0.00	6.25	128.00		
	2	900.00	2.50	2.60	0.00	6.50	138.46		
	3	700.00	2.50	2.50	0.00	6.25	112.00	126.15	13.33
Sulfur +5% DCP +50% Aggregate	1	950.00	2.50	2.50	0.00	6.25	152.00		
	2	900.00	2.50	2.50	0.00	6.25	144.00		
	3	850.00	2.50	2.50	0.00	6.25	136.00	144.00	8.00
Sulfur +5% DCP +50% Aggregate +1% Fiber Glass	1	800.00	2.50	2.50	0.00	6.25	128.00		
	2	800.00	2.50	2.50	0.00	6.25	128.00		
	3	850.00	2.50	2.50	0.00	6.25	136.00	130.67	4.62

APPENDIX B: STATISTICAL ANALYSES

The statistical analysis was performed using the Analysis of Variance (ANOVA) procedure with $\alpha=0.05$ to evaluate differences between the means of the dependent variables, the difference between the variable treatments results was evaluated using two methods; the Tukey's and pairwise t-tests

B-1: SAS Source Code to Run Compressive Strength Model

```
proc contents data=Work.strength; run;
  proc anova data=Work.strength;
/* class statement specifies the variables as classes*/
  class rep fg ag  material time;
/*
the model uses one strength to
run the proc ANOVA statement.
*/
  model strength=
  rep fg ag material fg*ag fg*ag*material time time*material time*ag
time*ag*material
time*fg time*fg*material time*ag*fg time*ag*fg*material;
run;

  means time/lsd tukey cldiff;
  means ag/lsd tukey cldiff;
  means fg/lsd tukey cldiff;
means material/lsd tukey cldiff;
run;
```

B-2: SAS Output for Compressive Strength Model

```
                                        The SAS System         12:22 Saturday, May 26,
2001  26

                                     The CONTENTS Procedure

        Data Set Name: WORK.DAMAGE                              Observations:        540
        Member Type:   DATA                                     Variables:           11
        Engine:        V8                                       Indexes:             0
        Created:       12:25 Saturday, May 26, 2001             Observation Length:  1080
        Last Modified: 12:25 Saturday, May 26, 2001             Deleted Observations: 0
        Protection:                                             Compressed:          NO
        Data Set Type:                                          Sorted:              NO
        Label:


                            -----Engine/Host Dependent Information-----

         Data Set Page Size:          16384
         Number of Data Set Pages:    37
         First Data Page:             1
         Max Obs per Page:            15
         Obs in First Data Page:      13
         Number of Data Set Repairs: 0
         File Name:                   d:\TEMP\SAS Temporary Files\_TD66\damage.sas7bdat
         Release Created:             8.0101M0
         Host Created:                WIN_NT


                         -----Alphabetic List of Variables and Attributes-----

                #    Variable     Type    Len    Pos    Format    Informat    Label
              ffffffffffffffffffffffffffffffffffffffffffffffffffffffffffffffffffffffff
                5    AG           Num       8     16                          AG
                1    Case         Num       8      0                          Case
                9    F9           Char    255    566    $255.     $255.       F9
               10    F10          Char    255    821    $255.     $255.       F10
               11    F11          Num       8     48                          F11
                4    FG           Num       8      8                          FG
                3    Material     Char    255    311    $255.     $255.       Material
                7    Rep          Num       8     32                          Rep
                8    STRENGTH     Num       8     40                          STRENGTH
                2    Sample_ID    Char    255     56    $255.     $255.       Sample_ID
                6    TIME         Num       8     24                          TIME
                                        The SAS System         12:22 Saturday, May 26,
2001  27

                                       The ANOVA Procedure

                                     Class Level Information

                     Class         Levels    Values

                     Rep                3    1 2 3

                     FG                 2    0 1

                     AG                 6    0 10 20 30 40 50

                     Material           3    Sulfur Sulfur_3% DCP Sulfur_5% DCP

                     TIME               5    1 24 72 168 336
```

```
                           Number of observations    540

                                   The SAS System        12:22 Saturday, May 26,
2001  28

                                The ANOVA Procedure

Dependent Variable: STRENGTH   STRENGTH

                                              Sum of
      Source                     DF          Squares     Mean Square    F Value    Pr > F

      Model                     181       2676781689        14788849     139.98    <.0001

      Error                     358         37821296          105646

      Corrected Total           539       2714602985


                     R-Square     Coeff Var      Root MSE    STRENGTH Mean

                     0.986067      6.824098      325.0324         4763.009


      Source                     DF          Anova SS     Mean Square    F Value    Pr > F

      Rep                         2            614120          307060       2.91    0.0560
      FG                          1         120534751       120534751    1140.93    <.0001
      AG                          5        1283355339       256671068    2429.54    <.0001
      Material                    2         252114266       126057133    1193.20    <.0001
      FG*AG                       5           8336117         1667223      15.78    <.0001
      FG*AG*Material             22         593710262        26986830     255.45    <.0001
      TIME                        4         189972725        47493181     449.55    <.0001
      Material*TIME               8          68748025         8593503      81.34    <.0001
      AG*TIME                    20          13822664          691133       6.54    <.0001
      AG*Material*TIME           50         130699653         2613993      24.74    <.0001
      FG*TIME                     4          36042998         9010749      85.29    <.0001
      FG*Material*TIME           10         478475602        47847560     452.90    <.0001
      FG*AG*TIME                 20          11698669          584933       5.54    <.0001
      FG*AG*Material*TIME        28                 0               0       0.00    1.0000
                                   The SAS System        12:22 Saturday, May 26,
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                                The ANOVA Procedure

                              t Tests (LSD) for STRENGTH

  NOTE: This test controls the Type I comparisonwise error rate, not the experimentwise
error
                                      rate.


                         Alpha                            0.05
                         Error Degrees of Freedom          358
                         Error Mean Square            105646.1
                         Critical Value of t           1.96661
                         Least Significant Difference   86.986


                Comparisons significant at the 0.05 level are indicated by ***.


                              Difference
```

```
   TIME             Between        95% Confidence
 Comparison          Means              Limits

 336   - 168          69.21        -17.77    156.20
 336   - 72           79.86         -7.12    166.85
 336   - 24          191.20        104.22    278.19  ***
 336   - 1          1559.95       1472.97   1646.94  ***
 168   - 336         -69.21       -156.20     17.77
 168   - 72           10.65        -76.34     97.63
 168   - 24          121.99         35.00    208.98  ***
 168   - 1          1490.74       1403.75   1577.73  ***
 72    - 336         -79.86       -166.85      7.12
 72    - 168         -10.65        -97.63     76.34
 72    - 24          111.34         24.36    198.33  ***
 72    - 1          1480.09       1393.11   1567.08  ***
 24    - 336        -191.20       -278.19   -104.22  ***
 24    - 168        -121.99       -208.98    -35.00  ***
 24    - 72         -111.34       -198.33    -24.36  ***
 24    - 1          1368.75       1281.76   1455.74  ***
 1     - 336       -1559.95      -1646.94  -1472.97  ***
 1     - 168       -1490.74      -1577.73  -1403.75  ***
 1     - 72        -1480.09      -1567.08  -1393.11  ***
 1     - 24        -1368.75      -1455.74  -1281.76  ***

                            The SAS System        12:22 Saturday, May 26,
2001  30

                         The ANOVA Procedure

             Tukey's Studentized Range (HSD) Test for STRENGTH

       NOTE: This test controls the Type I experimentwise error rate.


            Alpha                                   0.05
            Error Degrees of Freedom                 358
            Error Mean Square                   105646.1
            Critical Value of Studentized Range  3.87741
            Minimum Significant Difference        121.27


      Comparisons significant at the 0.05 level are indicated by ***.


                         Difference
   TIME                   Between         Simultaneous 95%
 Comparison                Means         Confidence Limits

 336   - 168          69.21        -52.06    190.48
 336   - 72           79.86        -41.41    201.13
 336   - 24          191.20         69.93    312.47  ***
 336   - 1          1559.95       1438.68   1681.22  ***
 168   - 336         -69.21       -190.48     52.06
 168   - 72           10.65       -110.62    131.92
 168   - 24          121.99          0.72    243.26  ***
 168   - 1          1490.74       1369.47   1612.01  ***
 72    - 336         -79.86       -201.13     41.41
 72    - 168         -10.65       -131.92    110.62
 72    - 24          111.34         -9.93    232.61
 72    - 1          1480.09       1358.82   1601.36  ***
 24    - 336        -191.20       -312.47    -69.93  ***
 24    - 168        -121.99       -243.26     -0.72  ***
 24    - 72         -111.34       -232.61      9.93
 24    - 1          1368.75       1247.48   1490.02  ***
 1     - 336       -1559.95      -1681.22  -1438.68  ***
 1     - 168       -1490.74      -1612.01  -1369.47  ***
```

```
                    1     - 72        -1480.09      -1601.36  -1358.82  ***
                    1     - 24        -1368.75      -1490.02  -1247.48  ***

                                         The SAS System         12:22 Saturday, May 26,
2001  31

                                       The ANOVA Procedure

                                    t Tests (LSD) for STRENGTH

   NOTE: This test controls the Type I comparisonwise error rate, not the experimentwise
error
                                              rate.

                                Alpha                           0.05
                                Error Degrees of Freedom         358
                                Error Mean Square           105646.1
                                Critical Value of t          1.96661
                                Least Significant Difference  95.288


                   Comparisons significant at the 0.05 level are indicated by ***.


                                   Difference
                         AG          Between       95% Confidence
                    Comparison        Means            Limits

                    50    - 40        592.22        496.93    687.51  ***
                    50    - 30       2097.50       2002.21   2192.79  ***
                    50    - 20       2509.44       2414.16   2604.73  ***
                    50    - 10       3544.44       3449.16   3639.73  ***
                    50    - 0        4418.33       4323.05   4513.62  ***
                    40    - 50       -592.22       -687.51   -496.93  ***
                    40    - 30       1505.28       1409.99   1600.57  ***
                    40    - 20       1917.22       1821.93   2012.51  ***
                    40    - 10       2952.22       2856.93   3047.51  ***
                    40    - 0        3826.11       3730.82   3921.40  ***
                    30    - 50      -2097.50      -2192.79  -2002.21  ***
                    30    - 40      -1505.28      -1600.57  -1409.99  ***
                    30    - 20        411.94        316.66    507.23  ***
                    30    - 10       1446.94       1351.66   1542.23  ***
                    30    - 0        2320.83       2225.55   2416.12  ***
                    20    - 50      -2509.44      -2604.73  -2414.16  ***
                    20    - 40      -1917.22      -2012.51  -1821.93  ***
                    20    - 30       -411.94       -507.23   -316.66  ***
                    20    - 10       1035.00        939.71   1130.29  ***
                    20    - 0        1908.89       1813.60   2004.18  ***
                    10    - 50      -3544.44      -3639.73  -3449.16  ***
                    10    - 40      -2952.22      -3047.51  -2856.93  ***
                    10    - 30      -1446.94      -1542.23  -1351.66  ***
                    10    - 20      -1035.00      -1130.29   -939.71  ***
                    10    - 0         873.89        778.60    969.18  ***
                    0     - 50      -4418.33      -4513.62  -4323.05  ***
                    0     - 40      -3826.11      -3921.40  -3730.82  ***
                    0     - 30      -2320.83      -2416.12  -2225.55  ***
                    0     - 20      -1908.89      -2004.18  -1813.60  ***
                    0     - 10       -873.89       -969.18   -778.60  ***

                                         The SAS System         12:22 Saturday, May 26,
2001  32

                                       The ANOVA Procedure

                             Tukey's Studentized Range (HSD) Test for STRENGTH
```

```
NOTE: This test controls the Type I experimentwise error rate.

                 Alpha                                   0.05
                 Error Degrees of Freedom                 358
                 Error Mean Square                   105646.1
                 Critical Value of Studentized Range  4.05205
                 Minimum Significant Difference        138.83

Comparisons significant at the 0.05 level are indicated by ***.

                          Difference
         AG                Between       Simultaneous 95%
     Comparison             Means       Confidence Limits

     50    - 40            592.22       453.39     731.05  ***
     50    - 30           2097.50      1958.67    2236.33  ***
     50    - 20           2509.44      2370.62    2648.27  ***
     50    - 10           3544.44      3405.62    3683.27  ***
     50    - 0            4418.33      4279.50    4557.16  ***
     40    - 50           -592.22      -731.05    -453.39  ***
     40    - 30           1505.28      1366.45    1644.11  ***
     40    - 20           1917.22      1778.39    2056.05  ***
     40    - 10           2952.22      2813.39    3091.05  ***
     40    - 0            3826.11      3687.28    3964.94  ***
     30    - 50          -2097.50     -2236.33   -1958.67  ***
     30    - 40          -1505.28     -1644.11   -1366.45  ***
     30    - 20            411.94       273.12     550.77  ***
     30    - 10           1446.94      1308.12    1585.77  ***
     30    - 0            2320.83      2182.00    2459.66  ***
     20    - 50          -2509.44     -2648.27   -2370.62  ***
     20    - 40          -1917.22     -2056.05   -1778.39  ***
     20    - 30           -411.94      -550.77    -273.12  ***
     20    - 10           1035.00       896.17    1173.83  ***
     20    - 0            1908.89      1770.06    2047.72  ***
     10    - 50          -3544.44     -3683.27   -3405.62  ***
     10    - 40          -2952.22     -3091.05   -2813.39  ***
     10    - 30          -1446.94     -1585.77   -1308.12  ***
     10    - 20          -1035.00     -1173.83    -896.17  ***
     10    - 0             873.89       735.06    1012.72  ***
     0     - 50          -4418.33     -4557.16   -4279.50  ***
     0     - 40          -3826.11     -3964.94   -3687.28  ***
     0     - 30          -2320.83     -2459.66   -2182.00  ***
     0     - 20          -1908.89     -2047.72   -1770.06  ***
     0     - 10           -873.89     -1012.72    -735.06  ***

                               The SAS System        12:22 Saturday, May 26,
2001  33

                                   The ANOVA Procedure

                                 t Tests (LSD) for STRENGTH

  NOTE: This test controls the Type I comparisonwise error rate, not the experimentwise
error
                                           rate.

                      Alpha                              0.05
                      Error Degrees of Freedom            358
                      Error Mean Square              105646.1
                      Critical Value of t             1.96661
                      Least Significant Difference     55.015
```

```
              Comparisons significant at the 0.05 level are indicated by ***.


                                 Difference
                     FG           Between       95% Confidence
                 Comparison        Means            Limits

                 1     - 0         944.91      889.89   999.92  ***
                 0     - 1        -944.91     -999.92  -889.89  ***

                                     The SAS System         12:22 Saturday, May 26,
2001  34

                                   The ANOVA Procedure

                       Tukey's Studentized Range (HSD) Test for STRENGTH

               NOTE: This test controls the Type I experimentwise error rate.


                         Alpha                                     0.05
                         Error Degrees of Freedom                   358
                         Error Mean Square                     105646.1
                         Critical Value of Studentized Range  2.78121
                         Minimum Significant Difference         55.015


              Comparisons significant at the 0.05 level are indicated by ***.


                                 Difference
                     FG           Between     Simultaneous 95%
                 Comparison        Means     Confidence Limits

                 1     - 0         944.91      889.89   999.92  ***
                 0     - 1        -944.91     -999.92  -889.89  ***

                                     The SAS System         12:22 Saturday, May 26,
2001  35

                                   The ANOVA Procedure

                                 t Tests (LSD) for STRENGTH

   NOTE: This test controls the Type I comparisonwise error rate, not the experimentwise
error
                                          rate.


                            Alpha                              0.05
                            Error Degrees of Freedom            358
                            Error Mean Square              105646.1
                            Critical Value of t             1.96661
                            Least Significant Difference     67.379


              Comparisons significant at the 0.05 level are indicated by ***.


                                          Difference
                     Material              Between        95% Confidence
                    Comparison              Means             Limits

           Sulfur_3% DCP - Sulfur          899.72        832.34    967.10   ***
```

```
Sulfur_3% DCP - Sulfur_5% DCP       1672.08      1604.70   1739.46  ***
Sulfur        - Sulfur_3% DCP       -899.72      -967.10   -832.34  ***
Sulfur        - Sulfur_5% DCP        772.36       704.98    839.74  ***
Sulfur_5% DCP - Sulfur_3% DCP      -1672.08     -1739.46  -1604.70  ***
Sulfur_5% DCP - Sulfur              -772.36      -839.74   -704.98  ***

                             The SAS System         12:22 Saturday, May 26,
2001  36

                                The ANOVA Procedure

                   Tukey's Studentized Range (HSD) Test for STRENGTH

              NOTE: This test controls the Type I experimentwise error rate.


                    Alpha                                   0.05
                    Error Degrees of Freedom                 358
                    Error Mean Square                   105646.1
                    Critical Value of Studentized Range  3.32836
                    Minimum Significant Difference        80.634


             Comparisons significant at the 0.05 level are indicated by ***.


                                   Difference
                 Material             Between      Simultaneous 95%
                Comparison              Means    Confidence Limits

Sulfur_3% DCP - Sulfur               899.72       819.09    980.36  ***
Sulfur_3% DCP - Sulfur_5% DCP       1672.08      1591.45   1752.72  ***
Sulfur        - Sulfur_3% DCP       -899.72      -980.36   -819.09  ***
Sulfur        - Sulfur_5% DCP        772.36       691.73    853.00  ***
Sulfur_5% DCP - Sulfur_3% DCP      -1672.08     -1752.72  -1591.45  ***
Sulfur_5% DCP - Sulfur              -772.36      -853.00   -691.73  ***
```

B-3: SAS Source Code to Run Bond Strength Model

```
proc contents data=Work.bond; run;
  proc anova data=work.bond;
/* class statement specifies the variables as classes*/
  class rep fg ag temperature material;
/*
the model uses bondstrength to
run the proc anova statement
*/
  model bond=
  rep fg|ag|material|temperature;
run;

  means temperature/lsd tukey cldiff;
  means ag/lsd tukey cldiff;
  means fg/lsd tukey cldiff;
means material/lsd tukey cldiff;
run;

      */
```

B-4: SAS Output for Bond Strength Model

```
                                         The SAS System         13:01 Saturday, May 26,
2001   2

                                     The CONTENTS Procedure

        Data Set Name: WORK.SHRINKAGE                         Observations:          54
        Member Type:   DATA                                   Variables:             11
        Engine:        V8                                     Indexes:               0
        Created:       13:02 Saturday, May 26, 2001           Observation Length:    1328
        Last Modified: 13:02 Saturday, May 26, 2001           Deleted Observations:  0
        Protection:                                           Compressed:            NO
        Data Set Type:                                        Sorted:                NO
        Label:


                          -----Engine/Host Dependent Information-----

       Data Set Page Size:          16384
       Number of Data Set Pages:    5
       First Data Page:             1
       Max Obs per Page:            12
       Obs in First Data Page:      10
       Number of Data Set Repairs:  0
       File Name:                   d:\TEMP\SAS Temporary Files\_TD306\shrinkage.sas7bdat
       Release Created:             8.0101M0
       Host Created:                WIN_NT


                     -----Alphabetic List of Variables and Attributes-----

          #    Variable       Type    Len    Pos    Format    Informat    Label
         fffffffffffffffffffffffffffffffffffffffffffffffffffffffffffffffffffffffffffff
          5    AG             Num       8     16                          AG
          1    Case           Num       8      0                          Case
          2    Case_ID        Char    255     48    $255.     $255.       Case_ID
          9    F9             Char    255    558    $255.     $255.       F9
         10    F10            Char    255    813    $255.     $255.       F10
         11    F11            Char    255   1068    $255.     $255.       F11
          4    FG             Num       8      8                          FG
          6    Temperature    Num       8     24                          Temperature
          8    bond           Num       8     40                          bond
          3    material       Char    255    303    $255.     $255.       material
          7    rep            Num       8     32                          rep
                                         The SAS System         13:01 Saturday, May 26,
2001   3

                                      The ANOVA Procedure

                                    Class Level Information

                  Class             Levels    Values

                  rep                    3    1 2 3

                  FG                     2    0 1

                  AG                     2    0 50

                  Temperature            2    270 290
```

```
             material              3    Sulfur Sulfur_3%DCP Sulfur_5%DCP


                               Number of observations     54

                                          The SAS System        13:01 Saturday, May 26,
2001   4

                                        The ANOVA Procedure

Dependent Variable: bond    bond

                                                 Sum of
      Source                      DF           Squares     Mean Square    F Value    Pr > F

      Model                       19       25892.90143      1362.78429       9.98    <.0001

      Error                       34        4644.67638       136.60813

      Corrected Total             53       30537.57782


                     R-Square     Coeff Var      Root MSE     bond Mean

                     0.847903      8.506981      11.68795      137.3924


      Source                      DF          Anova SS     Mean Square    F Value    Pr > F

      rep                          2       230.068621      115.034310       0.84    0.4396
      FG                           1       183.825898      183.825898       1.35    0.2541
      AG                           1      6911.264607     6911.264607      50.59    <.0001
      FG*AG                        0         0.000000          .             .         .
      material                     2      5837.080844     2918.540422      21.36    <.0001
      FG*material                  2       271.476829      135.738415       0.99    0.3807
      AG*material                  2      1435.742880      717.871440       5.25    0.0103
      FG*AG*material               0         0.000000          .             .         .
      Temperature                  1       353.838944      353.838944       2.59    0.1168
      FG*Temperature               1       135.169647      135.169647       0.99    0.3269
      AG*Temperature               1        31.624538       31.624538       0.23    0.6335
      FG*AG*Temperature            0         0.000000          .             .         .
      Temperature*material         2      2085.574046     1042.787023       7.63    0.0018
      FG*Temperat*material         2      4081.335284     2040.667642      14.94    <.0001
      AG*Temperat*material         2      2632.492114     1316.246057       9.64    0.0005
      FG*AG*Temper*materia         0         0.000000          .             .         .
                                          The SAS System        13:01 Saturday, May 26,
2001   5

                                        The ANOVA Procedure

                                       t Tests (LSD) for bond

  NOTE: This test controls the Type I comparisonwise error rate, not the experimentwise
error
                                              rate.


                              Alpha                            0.05
                              Error Degrees of Freedom           34
                              Error Mean Square            136.6081
                              Critical Value of t           2.03224
                              Least Significant Difference   6.4647


              Comparisons significant at the 0.05 level are indicated by ***.
```

```
                                      Difference
                       Temperature      Between      95% Confidence
                       Comparison         Means          Limits

                       290  - 270         5.120      -1.345  11.584
                       270  - 290        -5.120     -11.584   1.345

                                         The SAS System         13:01 Saturday, May 26,
2001   6

                                      The ANOVA Procedure

                         Tukey's Studentized Range (HSD) Test for bond

                 NOTE: This test controls the Type I experimentwise error rate.


                          Alpha                                   0.05
                          Error Degrees of Freedom                  34
                          Error Mean Square                   136.6081
                          Critical Value of Studentized Range  2.87405
                          Minimum Significant Difference        6.4647


                Comparisons significant at the 0.05 level are indicated by ***.


                                      Difference      Simultaneous
                       Temperature      Between      95% Confidence
                       Comparison         Means          Limits

                       290  - 270         5.120      -1.345  11.584
                       270  - 290        -5.120     -11.584   1.345

                                         The SAS System         13:01 Saturday, May 26,
2001   7

                                      The ANOVA Procedure

                                     t Tests (LSD) for bond

  NOTE: This test controls the Type I comparisonwise error rate, not the experimentwise
error
                                             rate.


                               Alpha                       0.05
                               Error Degrees of Freedom      34
                               Error Mean Square       136.6081
                               Critical Value of t      2.03224


                Comparisons significant at the 0.05 level are indicated by ***.


                                     Difference
                          AG           Between       95% Confidence
                      Comparison         Means           Limits

                      50    - 0         23.999       17.142   30.856  ***
                      0     - 50       -23.999      -30.856  -17.142  ***

                                         The SAS System         13:01 Saturday, May 26,
2001   8
```

```
                                      The ANOVA Procedure

                         Tukey's Studentized Range (HSD) Test for bond

                 NOTE: This test controls the Type I experimentwise error rate.


                          Alpha                                   0.05
                          Error Degrees of Freedom                  34
                          Error Mean Square                   136.6081
                          Critical Value of Studentized Range  2.87405


                Comparisons significant at the 0.05 level are indicated by ***.


                                     Difference
                          AG            Between     Simultaneous 95%
                      Comparison          Means    Confidence Limits

                      50    - 0          23.999      17.142   30.856  ***
                      0     - 50        -23.999     -30.856  -17.142  ***

                                         The SAS System         13:01 Saturday, May 26,
2001   9

                                      The ANOVA Procedure

                                     t Tests (LSD) for bond

  NOTE: This test controls the Type I comparisonwise error rate, not the experimentwise
error
                                             rate.


                               Alpha                            0.05
                               Error Degrees of Freedom           34
                               Error Mean Square            136.6081
                               Critical Value of t           2.03224


                Comparisons significant at the 0.05 level are indicated by ***.


                                      Difference
                           FG            Between      95% Confidence
                       Comparison          Means          Limits

                       0     - 1           3.914     -2.943  10.771
                       1     - 0          -3.914    -10.771   2.943

                                         The SAS System         13:01 Saturday, May 26,
2001  10

                                      The ANOVA Procedure

                         Tukey's Studentized Range (HSD) Test for bond

                 NOTE: This test controls the Type I experimentwise error rate.


                          Alpha                                   0.05
                          Error Degrees of Freedom                  34
                          Error Mean Square                   136.6081
                          Critical Value of Studentized Range  2.87405
```

```
                Comparisons significant at the 0.05 level are indicated by ***.


                                      Difference     Simultaneous
                           FG            Between    95% Confidence
                       Comparison          Means        Limits

                       0    - 1            3.914    -2.943  10.771
                       1    - 0           -3.914   -10.771   2.943

                                         The SAS System        13:01 Saturday, May 26,
2001  11

                                      The ANOVA Procedure

                                     t Tests (LSD) for bond

  NOTE: This test controls the Type I comparisonwise error rate, not the experimentwise
error
                                             rate.


                             Alpha                              0.05
                             Error Degrees of Freedom             34
                             Error Mean Square              136.6081
                             Critical Value of t             2.03224
                             Least Significant Difference     7.9176


                Comparisons significant at the 0.05 level are indicated by ***.


                                             Difference
                       material                 Between      95% Confidence
                      Comparison                  Means          Limits

              Sulfur_3%DCP - Sulfur_5%DCP        13.376       5.458   21.293  ***
              Sulfur_3%DCP - Sulfur              25.456      17.538   33.374  ***
              Sulfur_5%DCP - Sulfur_3%DCP       -13.376     -21.293   -5.458  ***
              Sulfur_5%DCP - Sulfur              12.080       4.163   19.998  ***
              Sulfur       - Sulfur_3%DCP       -25.456     -33.374  -17.538  ***
              Sulfur       - Sulfur_5%DCP       -12.080     -19.998   -4.163  ***

                                         The SAS System        13:01 Saturday, May 26,
2001  12

                                      The ANOVA Procedure

                         Tukey's Studentized Range (HSD) Test for bond

                 NOTE: This test controls the Type I experimentwise error rate.


                          Alpha                                     0.05
                          Error Degrees of Freedom                    34
                          Error Mean Square                     136.6081
                          Critical Value of Studentized Range    3.46544
                          Minimum Significant Difference          9.5469


                Comparisons significant at the 0.05 level are indicated by ***.


                                             Difference
```

```
        material               Between    Simultaneous 95%
       Comparison               Means    Confidence Limits

Sulfur_3%DCP - Sulfur_5%DCP    13.376       3.829   22.923  ***
Sulfur_3%DCP - Sulfur          25.456      15.909   35.003  ***
Sulfur_5%DCP - Sulfur_3%DCP   -13.376     -22.923   -3.829  ***
Sulfur_5%DCP - Sulfur          12.080       2.533   21.627  ***
Sulfur       - Sulfur_3%DCP   -25.456     -35.003  -15.909  ***
Sulfur       - Sulfur_5%DCP   -12.080     -21.627   -2.533  ***
```

B-5: SAS Source Code to Run Shrinkage Model

```
proc contents data=Work.shrinkage; run;
  proc anova data=work.shrinkage;
/* class statement specifies the variables as classes*/
  class rep fg ag  material time;
/*
the model uses shrinkage to
run the proc anova statement
*/
  model shrinkage=
  rep fg ag material fg*ag fg*ag*material time time*material time*ag
time*ag*material
time*fg time*fg*material time*ag*fg time*ag*fg*material;
run;
/*
random
  rep*fg
  rep*ag
  rep*material
rep*ag*fg
rep*ag*fg*material;
lsmeans fg;
lsmeans ag;
lsmeans time;
lsmeans material;
*/
  means time/lsd tukey cldiff;
  means ag/lsd tukey cldiff;
  means fg/lsd tukey cldiff;
means material/lsd tukey cldiff;
run;

      */
```

B-4: SAS Output for Bond Strength Model

```
                                         The SAS System        12:56 Saturday, May 26,
2001   1

                                     The CONTENTS Procedure

         Data Set Name: WORK.DAMAGE                           Observations:          306
         Member Type:   DATA                                  Variables:             8
         Engine:        V8                                    Indexes:               0
         Created:       12:57 Saturday, May 26, 2001          Observation Length:    560
         Last Modified: 12:57 Saturday, May 26, 2001          Deleted Observations:  0
         Protection:                                          Compressed:            NO
         Data Set Type:                                       Sorted:                NO
         Label:


                          -----Engine/Host Dependent Information-----

         Data Set Page Size:          16384
         Number of Data Set Pages:    11
         First Data Page:             1
         Max Obs per Page:            29
         Obs in First Data Page:      25
         Number of Data Set Repairs:  0
         File Name:                   d:\TEMP\SAS Temporary Files\_TD275\damage.sas7bdat
         Release Created:             8.0101M0
         Host Created:                WIN_NT


                     -----Alphabetic List of Variables and Attributes-----

            #    Variable     Type    Len    Pos    Format    Informat    Label
            ffffffffffffffffffffffffffffffffffffffffffffffffffffffffffffffffffffff
            5    Ag           Num       8     16                          Ag
            1    Case         Num       8      0                          Case
            2    Case_ID      Char    255     48    $255.     $255.       Case_ID
            4    FG           Num       8      8                          FG
            3    Material     Char    255    303    $255.     $255.       Material
            7    rep          Num       8     32                          rep
            8    shrinkage    Num       8     40                          shrinkage
            6    time         Num       8     24                          time
                                         The SAS System        12:56 Saturday, May 26,
2001   2

                                      The ANOVA Procedure

                                    Class Level Information

Class       Levels  Values

rep              2  1 2

FG               2  0 1

Ag               2  0 50

Material         3  Sulfur Sulfur_3%DCP Sulfur_5%DCP

time            19  0.15 0.5 0.6666666667 0.67 0.83 0.8333333333 1 1.5 2 2.5 3 4 8 24 48
72 96
                    120 144
```

```
                                   Number of observations    306
                                           The SAS System        12:56 Saturday, May 26,
2001   3

                                        The ANOVA Procedure

Dependent Variable: shrinkage   shrinkage

                                                    Sum of
        Source                      DF             Squares     Mean Square    F Value    Pr > F

        Model                      171          3.12033098      0.01824755     181.67    <.0001

        Error                      134          0.01345971      0.00010045

        Corrected Total            305          3.13379069


                       R-Square     Coeff Var      Root MSE    shrinkage Mean

                       0.995705      4.314341      0.010022          0.232301


        Source                      DF            Anova SS     Mean Square    F Value    Pr > F

        rep                          1          0.00200919      0.00200919      20.00    <.0001
        FG                           1          0.48093426      0.48093426    4788.01    <.0001
        Ag                           1          0.80144637      0.80144637    7978.91    <.0001
        Material                     2          1.15386221      0.57693111    5743.72    <.0001
        FG*Ag                        0          0.00000000           .            .         .
        FG*Ag*Material               4          0.21373145      0.05343286     531.96    <.0001
        time                        18          0.47990089      0.02666116     265.43    <.0001
        Material*time               36          0.05409428      0.00150262      14.96    <.0001
        Ag*time                     18          0.23003575      0.01277976     127.23    <.0001
        Ag*Material*time            38          0.21409808      0.00563416      56.09    <.0001
        FG*time                     18          0.05007573      0.00278198      27.70    <.0001
        FG*Material*time            38          0.06530301      0.00171850      17.11    <.0001
        FG*Ag*time                   0          0.00000000           .            .         .
        FG*Ag*Material*time         -4          0.00000000      0.00000000       0.00    <.0001
                                           The SAS System        12:56 Saturday, May 26,
2001   4

                                        The ANOVA Procedure

                                     t Tests (LSD) for shrinkage

  NOTE: This test controls the Type I comparisonwise error rate, not the experimentwise
error
                                                rate.


                                  Alpha                         0.05
                                  Error Degrees of Freedom       134
                                  Error Mean Square           0.0001
                                  Critical Value of t        1.97783


                    Comparisons significant at the 0.05 level are indicated by ***.


                                                 Difference
                            time                    Between       95% Confidence
                         Comparison                   Means           Limits
```

```
120          - 72               0.001000    -0.005607  0.007607
120          - 144              0.001000    -0.005607  0.007607
120          - 96               0.001222    -0.005385  0.007830
120          - 48               0.004667    -0.001941  0.011274
120          - 24               0.009444     0.002837  0.016052  ***
120          - 8                0.027222     0.020615  0.033830  ***
120          - 4                0.049611     0.043004  0.056219  ***
120          - 3                0.052333     0.045726  0.058941  ***
120          - 2.5              0.053833     0.047226  0.060441  ***
120          - 2                0.055278     0.048670  0.061885  ***
120          - 1.5              0.057000     0.050393  0.063607  ***
120          - 1                0.064106     0.057498  0.070713  ***
120          - 0.8333333333     0.068056     0.059963  0.076148  ***
120          - 0.83             0.075722     0.067630  0.083815  ***
120          - 0.6666666667     0.081611     0.073519  0.089704  ***
120          - 0.67             0.088833     0.080741  0.096926  ***
120          - 0.5              0.103889     0.097281  0.110496  ***
120          - 0.15             0.140111     0.133504  0.146719  ***
72           - 120             -0.001000    -0.007607  0.005607
72           - 144              0.000000    -0.006607  0.006607
72           - 96               0.000222    -0.006385  0.006830
72           - 48               0.003667    -0.002941  0.010274
72           - 24               0.008444     0.001837  0.015052  ***
72           - 8                0.026222     0.019615  0.032830  ***
72           - 4                0.048611     0.042004  0.055219  ***
72           - 3                0.051333     0.044726  0.057941  ***
72           - 2.5              0.052833     0.046226  0.059441  ***
72           - 2                0.054278     0.047670  0.060885  ***
72           - 1.5              0.056000     0.049393  0.062607  ***
72           - 1                0.063106     0.056498  0.069713  ***
72           - 0.8333333333     0.067056     0.058963  0.075148  ***

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                                  The ANOVA Procedure

                               t Tests (LSD) for shrinkage

          Comparisons significant at the 0.05 level are indicated by ***.


                                Difference
                 time             Between        95% Confidence
              Comparison           Means             Limits

72           - 0.83             0.074722     0.066630  0.082815  ***
72           - 0.6666666667     0.080611     0.072519  0.088704  ***
72           - 0.67             0.087833     0.079741  0.095926  ***
72           - 0.5              0.102889     0.096281  0.109496  ***
72           - 0.15             0.139111     0.132504  0.145719  ***
144          - 120             -0.001000    -0.007607  0.005607
144          - 72              -0.000000    -0.006607  0.006607
144          - 96               0.000222    -0.006385  0.006830
144          - 48               0.003667    -0.002941  0.010274
144          - 24               0.008444     0.001837  0.015052  ***
144          - 8                0.026222     0.019615  0.032830  ***
144          - 4                0.048611     0.042004  0.055219  ***
144          - 3                0.051333     0.044726  0.057941  ***
144          - 2.5              0.052833     0.046226  0.059441  ***
144          - 2                0.054278     0.047670  0.060885  ***
144          - 1.5              0.056000     0.049393  0.062607  ***
144          - 1                0.063106     0.056498  0.069713  ***
144          - 0.8333333333     0.067056     0.058963  0.075148  ***
144          - 0.83             0.074722     0.066630  0.082815  ***
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           144           - 0.6666666667      0.080611      0.072519  0.088704  ***
           144           - 0.67              0.087833      0.079741  0.095926  ***
           144           - 0.5               0.102889      0.096281  0.109496  ***
           144           - 0.15              0.139111      0.132504  0.145719  ***
           96            - 120              -0.001222     -0.007830  0.005385
           96            - 72               -0.000222     -0.006830  0.006385
           96            - 144              -0.000222     -0.006830  0.006385
           96            - 48                0.003444     -0.003163  0.010052
           96            - 24                0.008222      0.001615  0.014830  ***
           96            - 8                 0.026000      0.019393  0.032607  ***
           96            - 4                 0.048389      0.041781  0.054996  ***
           96            - 3                 0.051111      0.044504  0.057719  ***
           96            - 2.5               0.052611      0.046004  0.059219  ***
           96            - 2                 0.054056      0.047448  0.060663  ***
           96            - 1.5               0.055778      0.049170  0.062385  ***
           96            - 1                 0.062883      0.056276  0.069491  ***
           96            - 0.8333333333      0.066833      0.058741  0.074926  ***
           96            - 0.83              0.074500      0.066408  0.082592  ***
           96            - 0.6666666667      0.080389      0.072296  0.088481  ***
           96            - 0.67              0.087611      0.079519  0.095704  ***
           96            - 0.5               0.102667      0.096059  0.109274  ***
           96            - 0.15              0.138889      0.132281  0.145496  ***

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                                  The ANOVA Procedure

                               t Tests (LSD) for shrinkage

                Comparisons significant at the 0.05 level are indicated by ***.


                                            Difference
                        time                 Between       95% Confidence
                     Comparison               Means            Limits

           48            - 120              -0.004667     -0.011274  0.001941
           48            - 72               -0.003667     -0.010274  0.002941
           48            - 144              -0.003667     -0.010274  0.002941
           48            - 96               -0.003444     -0.010052  0.003163
           48            - 24                0.004778     -0.001830  0.011385
           48            - 8                 0.022556      0.015948  0.029163  ***
           48            - 4                 0.044944      0.038337  0.051552  ***
           48            - 3                 0.047667      0.041059  0.054274  ***
           48            - 2.5               0.049167      0.042559  0.055774  ***
           48            - 2                 0.050611      0.044004  0.057219  ***
           48            - 1.5               0.052333      0.045726  0.058941  ***
           48            - 1                 0.059439      0.052831  0.066046  ***
           48            - 0.8333333333      0.063389      0.055296  0.071481  ***
           48            - 0.83              0.071056      0.062963  0.079148  ***
           48            - 0.6666666667      0.076944      0.068852  0.085037  ***
           48            - 0.67              0.084167      0.076074  0.092259  ***
           48            - 0.5               0.099222      0.092615  0.105830  ***
           48            - 0.15              0.135444      0.128837  0.142052  ***
           24            - 120              -0.009444     -0.016052 -0.002837  ***
           24            - 72               -0.008444     -0.015052 -0.001837  ***
           24            - 144              -0.008444     -0.015052 -0.001837  ***
           24            - 96               -0.008222     -0.014830 -0.001615  ***
           24            - 48               -0.004778     -0.011385  0.001830
           24            - 8                 0.017778      0.011170  0.024385  ***
           24            - 4                 0.040167      0.033559  0.046774  ***
           24            - 3                 0.042889      0.036281  0.049496  ***
           24            - 2.5               0.044389      0.037781  0.050996  ***
           24            - 2                 0.045833      0.039226  0.052441  ***
           24            - 1.5               0.047556      0.040948  0.054163  ***
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24          - 1                  0.054661     0.048054  0.061269  ***
24          - 0.8333333333       0.058611     0.050519  0.066704  ***
24          - 0.83               0.066278     0.058185  0.074370  ***
24          - 0.6666666667       0.072167     0.064074  0.080259  ***
24          - 0.67               0.079389     0.071296  0.087481  ***
24          - 0.5                0.094444     0.087837  0.101052  ***
24          - 0.15               0.130667     0.124059  0.137274  ***
8           - 120               -0.027222    -0.033830 -0.020615  ***
8           - 72                -0.026222    -0.032830 -0.019615  ***
8           - 144               -0.026222    -0.032830 -0.019615  ***
8           - 96                -0.026000    -0.032607 -0.019393  ***
8           - 48                -0.022556    -0.029163 -0.015948  ***

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                              The ANOVA Procedure

                          t Tests (LSD) for shrinkage

            Comparisons significant at the 0.05 level are indicated by ***.


                                Difference
                 time             Between        95% Confidence
              Comparison           Means             Limits

8           - 24                -0.017778    -0.024385 -0.011170  ***
8           - 4                  0.022389     0.015781  0.028996  ***
8           - 3                  0.025111     0.018504  0.031719  ***
8           - 2.5                0.026611     0.020004  0.033219  ***
8           - 2                  0.028056     0.021448  0.034663  ***
8           - 1.5                0.029778     0.023170  0.036385  ***
8           - 1                  0.036883     0.030276  0.043491  ***
8           - 0.8333333333       0.040833     0.032741  0.048926  ***
8           - 0.83               0.048500     0.040408  0.056592  ***
8           - 0.6666666667       0.054389     0.046296  0.062481  ***
8           - 0.67               0.061611     0.053519  0.069704  ***
8           - 0.5                0.076667     0.070059  0.083274  ***
8           - 0.15               0.112889     0.106281  0.119496  ***
4           - 120               -0.049611    -0.056219 -0.043004  ***
4           - 72                -0.048611    -0.055219 -0.042004  ***
4           - 144               -0.048611    -0.055219 -0.042004  ***
4           - 96                -0.048389    -0.054996 -0.041781  ***
4           - 48                -0.044944    -0.051552 -0.038337  ***
4           - 24                -0.040167    -0.046774 -0.033559  ***
4           - 8                 -0.022389    -0.028996 -0.015781  ***
4           - 3                  0.002722    -0.003885  0.009330
4           - 2.5                0.004222    -0.002385  0.010830
4           - 2                  0.005667    -0.000941  0.012274
4           - 1.5                0.007389     0.000781  0.013996  ***
4           - 1                  0.014494     0.007887  0.021102  ***
4           - 0.8333333333       0.018444     0.010352  0.026537  ***
4           - 0.83               0.026111     0.018019  0.034204  ***
4           - 0.6666666667       0.032000     0.023908  0.040092  ***
4           - 0.67               0.039222     0.031130  0.047315  ***
4           - 0.5                0.054278     0.047670  0.060885  ***
4           - 0.15               0.090500     0.083893  0.097107  ***
3           - 120               -0.052333    -0.058941 -0.045726  ***
3           - 72                -0.051333    -0.057941 -0.044726  ***
3           - 144               -0.051333    -0.057941 -0.044726  ***
3           - 96                -0.051111    -0.057719 -0.044504  ***
3           - 48                -0.047667    -0.054274 -0.041059  ***
3           - 24                -0.042889    -0.049496 -0.036281  ***
3           - 8                 -0.025111    -0.031719 -0.018504  ***
3           - 4                 -0.002722    -0.009330  0.003885
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              3            - 2.5             0.001500    -0.005107  0.008107
              3            - 2               0.002944    -0.003663  0.009552

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                                     The ANOVA Procedure

                                  t Tests (LSD) for shrinkage

                  Comparisons significant at the 0.05 level are indicated by ***.


                                            Difference
                          time               Between        95% Confidence
                        Comparison            Means             Limits

              3            - 1.5             0.004667    -0.001941  0.011274
              3            - 1               0.011772     0.005165  0.018380  ***
              3            - 0.8333333333    0.015722     0.007630  0.023815  ***
              3            - 0.83            0.023389     0.015296  0.031481  ***
              3            - 0.6666666667    0.029278     0.021185  0.037370  ***
              3            - 0.67            0.036500     0.028408  0.044592  ***
              3            - 0.5             0.051556     0.044948  0.058163  ***
              3            - 0.15            0.087778     0.081170  0.094385  ***
              2.5          - 120            -0.053833    -0.060441 -0.047226  ***
              2.5          - 72             -0.052833    -0.059441 -0.046226  ***
              2.5          - 144            -0.052833    -0.059441 -0.046226  ***
              2.5          - 96             -0.052611    -0.059219 -0.046004  ***
              2.5          - 48             -0.049167    -0.055774 -0.042559  ***
              2.5          - 24             -0.044389    -0.050996 -0.037781  ***
              2.5          - 8              -0.026611    -0.033219 -0.020004  ***
              2.5          - 4              -0.004222    -0.010830  0.002385
              2.5          - 3              -0.001500    -0.008107  0.005107
              2.5          - 2               0.001444    -0.005163  0.008052
              2.5          - 1.5             0.003167    -0.003441  0.009774
              2.5          - 1               0.010272     0.003665  0.016880  ***
              2.5          - 0.8333333333    0.014222     0.006130  0.022315  ***
              2.5          - 0.83            0.021889     0.013796  0.029981  ***
              2.5          - 0.6666666667    0.027778     0.019685  0.035870  ***
              2.5          - 0.67            0.035000     0.026908  0.043092  ***
              2.5          - 0.5             0.050056     0.043448  0.056663  ***
              2.5          - 0.15            0.086278     0.079670  0.092885  ***
              2            - 120            -0.055278    -0.061885 -0.048670  ***
              2            - 72             -0.054278    -0.060885 -0.047670  ***
              2            - 144            -0.054278    -0.060885 -0.047670  ***
              2            - 96             -0.054056    -0.060663 -0.047448  ***
              2            - 48             -0.050611    -0.057219 -0.044004  ***
              2            - 24             -0.045833    -0.052441 -0.039226  ***
              2            - 8              -0.028056    -0.034663 -0.021448  ***
              2            - 4              -0.005667    -0.012274  0.000941
              2            - 3              -0.002944    -0.009552  0.003663
              2            - 2.5            -0.001444    -0.008052  0.005163
              2            - 1.5             0.001722    -0.004885  0.008330
              2            - 1               0.008828     0.002220  0.015435  ***
              2            - 0.8333333333    0.012778     0.004685  0.020870  ***
              2            - 0.83            0.020444     0.012352  0.028537  ***
              2            - 0.6666666667    0.026333     0.018241  0.034426  ***

                                      The SAS System        12:56 Saturday, May 26,
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                                     The ANOVA Procedure

                                  t Tests (LSD) for shrinkage
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              Comparisons significant at the 0.05 level are indicated by ***.


                                        Difference
                    time                 Between       95% Confidence
                 Comparison               Means            Limits

          2            - 0.67             0.033556     0.025463  0.041648  ***
          2            - 0.5              0.048611     0.042004  0.055219  ***
          2            - 0.15             0.084833     0.078226  0.091441  ***
          1.5          - 120             -0.057000    -0.063607 -0.050393  ***
          1.5          - 72              -0.056000    -0.062607 -0.049393  ***
          1.5          - 144             -0.056000    -0.062607 -0.049393  ***
          1.5          - 96              -0.055778    -0.062385 -0.049170  ***
          1.5          - 48              -0.052333    -0.058941 -0.045726  ***
          1.5          - 24              -0.047556    -0.054163 -0.040948  ***
          1.5          - 8               -0.029778    -0.036385 -0.023170  ***
          1.5          - 4               -0.007389    -0.013996 -0.000781  ***
          1.5          - 3               -0.004667    -0.011274  0.001941
          1.5          - 2.5             -0.003167    -0.009774  0.003441
          1.5          - 2               -0.001722    -0.008330  0.004885
          1.5          - 1                0.007106     0.000498  0.013713  ***
          1.5          - 0.8333333333     0.011056     0.002963  0.019148  ***
          1.5          - 0.83             0.018722     0.010630  0.026815  ***
          1.5          - 0.6666666667     0.024611     0.016519  0.032704  ***
          1.5          - 0.67             0.031833     0.023741  0.039926  ***
          1.5          - 0.5              0.046889     0.040281  0.053496  ***
          1.5          - 0.15             0.083111     0.076504  0.089719  ***
          1            - 120             -0.064106    -0.070713 -0.057498  ***
          1            - 72              -0.063106    -0.069713 -0.056498  ***
          1            - 144             -0.063106    -0.069713 -0.056498  ***
          1            - 96              -0.062883    -0.069491 -0.056276  ***
          1            - 48              -0.059439    -0.066046 -0.052831  ***
          1            - 24              -0.054661    -0.061269 -0.048054  ***
          1            - 8               -0.036883    -0.043491 -0.030276  ***
          1            - 4               -0.014494    -0.021102 -0.007887  ***
          1            - 3               -0.011772    -0.018380 -0.005165  ***
          1            - 2.5             -0.010272    -0.016880 -0.003665  ***
          1            - 2               -0.008828    -0.015435 -0.002220  ***
          1            - 1.5             -0.007106    -0.013713 -0.000498  ***
          1            - 0.8333333333     0.003950    -0.004142  0.012042
          1            - 0.83             0.011617     0.003524  0.019709  ***
          1            - 0.6666666667     0.017506     0.009413  0.025598  ***
          1            - 0.67             0.024728     0.016635  0.032820  ***
          1            - 0.5              0.039783     0.033176  0.046391  ***
          1            - 0.15             0.076006     0.069398  0.082613  ***
          0.8333333333 - 120             -0.068056    -0.076148 -0.059963  ***
          0.8333333333 - 72              -0.067056    -0.075148 -0.058963  ***

                                    The SAS System        12:56 Saturday, May 26,
 2001  10

                                  The ANOVA Procedure

                                t Tests (LSD) for shrinkage

              Comparisons significant at the 0.05 level are indicated by ***.


                                        Difference
                    time                 Between       95% Confidence
                 Comparison               Means            Limits

          0.8333333333 - 144             -0.067056    -0.075148 -0.058963  ***
          0.8333333333 - 96              -0.066833    -0.074926 -0.058741  ***
          0.8333333333 - 48              -0.063389    -0.071481 -0.055296  ***
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0.8333333333 - 24              -0.058611    -0.066704 -0.050519  ***
0.8333333333 - 8               -0.040833    -0.048926 -0.032741  ***
0.8333333333 - 4               -0.018444    -0.026537 -0.010352  ***
0.8333333333 - 3               -0.015722    -0.023815 -0.007630  ***
0.8333333333 - 2.5             -0.014222    -0.022315 -0.006130  ***
0.8333333333 - 2               -0.012778    -0.020870 -0.004685  ***
0.8333333333 - 1.5             -0.011056    -0.019148 -0.002963  ***
0.8333333333 - 1               -0.003950    -0.012042  0.004142
0.8333333333 - 0.83             0.007667    -0.001678  0.017011
0.8333333333 - 0.6666666667     0.013556     0.004211  0.022900  ***
0.8333333333 - 0.67             0.020778     0.011433  0.030122  ***
0.8333333333 - 0.5              0.035833     0.027741  0.043926  ***
0.8333333333 - 0.15             0.072056     0.063963  0.080148  ***
0.83         - 120             -0.075722    -0.083815 -0.067630  ***
0.83         - 72              -0.074722    -0.082815 -0.066630  ***
0.83         - 144             -0.074722    -0.082815 -0.066630  ***
0.83         - 96              -0.074500    -0.082592 -0.066408  ***
0.83         - 48              -0.071056    -0.079148 -0.062963  ***
0.83         - 24              -0.066278    -0.074370 -0.058185  ***
0.83         - 8               -0.048500    -0.056592 -0.040408  ***
0.83         - 4               -0.026111    -0.034204 -0.018019  ***
0.83         - 3               -0.023389    -0.031481 -0.015296  ***
0.83         - 2.5             -0.021889    -0.029981 -0.013796  ***
0.83         - 2               -0.020444    -0.028537 -0.012352  ***
0.83         - 1.5             -0.018722    -0.026815 -0.010630  ***
0.83         - 1               -0.011617    -0.019709 -0.003524  ***
0.83         - 0.8333333333    -0.007667    -0.017011  0.001678
0.83         - 0.6666666667     0.005889    -0.003455  0.015233
0.83         - 0.67             0.013111     0.003767  0.022455  ***
0.83         - 0.5              0.028167     0.020074  0.036259  ***
0.83         - 0.15             0.064389     0.056296  0.072481  ***
0.6666666667 - 120             -0.081611    -0.089704 -0.073519  ***
0.6666666667 - 72              -0.080611    -0.088704 -0.072519  ***
0.6666666667 - 144             -0.080611    -0.088704 -0.072519  ***
0.6666666667 - 96              -0.080389    -0.088481 -0.072296  ***
0.6666666667 - 48              -0.076944    -0.085037 -0.068852  ***
0.6666666667 - 24              -0.072167    -0.080259 -0.064074  ***
0.6666666667 - 8               -0.054389    -0.062481 -0.046296  ***

                                 The SAS System         12:56 Saturday, May 26,
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                              The ANOVA Procedure

                          t Tests (LSD) for shrinkage

        Comparisons significant at the 0.05 level are indicated by ***.


                                 Difference
              time                 Between        95% Confidence
           Comparison               Means             Limits

0.6666666667 - 4               -0.032000    -0.040092 -0.023908  ***
0.6666666667 - 3               -0.029278    -0.037370 -0.021185  ***
0.6666666667 - 2.5             -0.027778    -0.035870 -0.019685  ***
0.6666666667 - 2               -0.026333    -0.034426 -0.018241  ***
0.6666666667 - 1.5             -0.024611    -0.032704 -0.016519  ***
0.6666666667 - 1               -0.017506    -0.025598 -0.009413  ***
0.6666666667 - 0.8333333333    -0.013556    -0.022900 -0.004211  ***
0.6666666667 - 0.83            -0.005889    -0.015233  0.003455
0.6666666667 - 0.67             0.007222    -0.002122  0.016567
0.6666666667 - 0.5              0.022278     0.014185  0.030370  ***
0.6666666667 - 0.15             0.058500     0.050408  0.066592  ***
0.67         - 120             -0.088833    -0.096926 -0.080741  ***
0.67         - 72              -0.087833    -0.095926 -0.079741  ***
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0.67          - 144            -0.087833    -0.095926 -0.079741  ***
0.67          - 96             -0.087611    -0.095704 -0.079519  ***
0.67          - 48             -0.084167    -0.092259 -0.076074  ***
0.67          - 24             -0.079389    -0.087481 -0.071296  ***
0.67          - 8              -0.061611    -0.069704 -0.053519  ***
0.67          - 4              -0.039222    -0.047315 -0.031130  ***
0.67          - 3              -0.036500    -0.044592 -0.028408  ***
0.67          - 2.5            -0.035000    -0.043092 -0.026908  ***
0.67          - 2              -0.033556    -0.041648 -0.025463  ***
0.67          - 1.5            -0.031833    -0.039926 -0.023741  ***
0.67          - 1              -0.024728    -0.032820 -0.016635  ***
0.67          - 0.8333333333   -0.020778    -0.030122 -0.011433  ***
0.67          - 0.83           -0.013111    -0.022455 -0.003767  ***
0.67          - 0.6666666667   -0.007222    -0.016567  0.002122
0.67          - 0.5             0.015056     0.006963  0.023148  ***
0.67          - 0.15            0.051278     0.043185  0.059370  ***
0.5           - 120            -0.103889    -0.110496 -0.097281  ***
0.5           - 72             -0.102889    -0.109496 -0.096281  ***
0.5           - 144            -0.102889    -0.109496 -0.096281  ***
0.5           - 96             -0.102667    -0.109274 -0.096059  ***
0.5           - 48             -0.099222    -0.105830 -0.092615  ***
0.5           - 24             -0.094444    -0.101052 -0.087837  ***
0.5           - 8              -0.076667    -0.083274 -0.070059  ***
0.5           - 4              -0.054278    -0.060885 -0.047670  ***
0.5           - 3              -0.051556    -0.058163 -0.044948  ***
0.5           - 2.5            -0.050056    -0.056663 -0.043448  ***
0.5           - 2              -0.048611    -0.055219 -0.042004  ***
0.5           - 1.5            -0.046889    -0.053496 -0.040281  ***

                             The SAS System         12:56 Saturday, May 26,
2001  12

                          The ANOVA Procedure

                        t Tests (LSD) for shrinkage

           Comparisons significant at the 0.05 level are indicated by ***.


                                 Difference
                 time             Between       95% Confidence
              Comparison           Means            Limits

0.5           - 1              -0.039783    -0.046391 -0.033176  ***
0.5           - 0.8333333333   -0.035833    -0.043926 -0.027741  ***
0.5           - 0.83           -0.028167    -0.036259 -0.020074  ***
0.5           - 0.6666666667   -0.022278    -0.030370 -0.014185  ***
0.5           - 0.67           -0.015056    -0.023148 -0.006963  ***
0.5           - 0.15            0.036222     0.029615  0.042830  ***
0.15          - 120            -0.140111    -0.146719 -0.133504  ***
0.15          - 72             -0.139111    -0.145719 -0.132504  ***
0.15          - 144            -0.139111    -0.145719 -0.132504  ***
0.15          - 96             -0.138889    -0.145496 -0.132281  ***
0.15          - 48             -0.135444    -0.142052 -0.128837  ***
0.15          - 24             -0.130667    -0.137274 -0.124059  ***
0.15          - 8              -0.112889    -0.119496 -0.106281  ***
0.15          - 4              -0.090500    -0.097107 -0.083893  ***
0.15          - 3              -0.087778    -0.094385 -0.081170  ***
0.15          - 2.5            -0.086278    -0.092885 -0.079670  ***
0.15          - 2              -0.084833    -0.091441 -0.078226  ***
0.15          - 1.5            -0.083111    -0.089719 -0.076504  ***
0.15          - 1              -0.076006    -0.082613 -0.069398  ***
0.15          - 0.8333333333   -0.072056    -0.080148 -0.063963  ***
0.15          - 0.83           -0.064389    -0.072481 -0.056296  ***
0.15          - 0.6666666667   -0.058500    -0.066592 -0.050408  ***
0.15          - 0.67           -0.051278    -0.059370 -0.043185  ***
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          0.15        - 0.5            -0.036222    -0.042830 -0.029615  ***

                                    The SAS System       12:56 Saturday, May 26,
2001  13

                                  The ANOVA Procedure

                      Tukey's Studentized Range (HSD) Test for shrinkage

                  NOTE: This test controls the Type I experimentwise error rate.

                        Alpha                                   0.05
                        Error Degrees of Freedom                 134
                        Error Mean Square                     0.0001
                        Critical Value of Studentized Range  5.07420


                 Comparisons significant at the 0.05 level are indicated by ***.


                                   Difference
                  time               Between     Simultaneous 95%
               Comparison              Means     Confidence Limits

          120         - 72             0.001000    -0.010987  0.012987
          120         - 144            0.001000    -0.010987  0.012987
          120         - 96             0.001222    -0.010764  0.013209
          120         - 48             0.004667    -0.007320  0.016653
          120         - 24             0.009444    -0.002542  0.021431
          120         - 8              0.027222     0.015236  0.039209  ***
          120         - 4              0.049611     0.037624  0.061598  ***
          120         - 3              0.052333     0.040347  0.064320  ***
          120         - 2.5            0.053833     0.041847  0.065820  ***
          120         - 2              0.055278     0.043291  0.067264  ***
          120         - 1.5            0.057000     0.045013  0.068987  ***
          120         - 1              0.064106     0.052119  0.076092  ***
          120         - 0.8333333333   0.068056     0.053375  0.082736  ***
          120         - 0.83           0.075722     0.061042  0.090403  ***
          120         - 0.6666666667   0.081611     0.066931  0.096292  ***
          120         - 0.67           0.088833     0.074153  0.103514  ***
          120         - 0.5            0.103889     0.091902  0.115876  ***
          120         - 0.15           0.140111     0.128124  0.152098  ***
          72          - 120           -0.001000    -0.012987  0.010987
          72          - 144            0.000000    -0.011987  0.011987
          72          - 96             0.000222    -0.011764  0.012209
          72          - 48             0.003667    -0.008320  0.015653
          72          - 24             0.008444    -0.003542  0.020431
          72          - 8              0.026222     0.014236  0.038209  ***
          72          - 4              0.048611     0.036624  0.060598  ***
          72          - 3              0.051333     0.039347  0.063320  ***
          72          - 2.5            0.052833     0.040847  0.064820  ***
          72          - 2              0.054278     0.042291  0.066264  ***
          72          - 1.5            0.056000     0.044013  0.067987  ***
          72          - 1              0.063106     0.051119  0.075092  ***
          72          - 0.8333333333   0.067056     0.052375  0.081736  ***
          72          - 0.83           0.074722     0.060042  0.089403  ***

                                    The SAS System       12:56 Saturday, May 26,
2001  14

                                  The ANOVA Procedure

                      Tukey's Studentized Range (HSD) Test for shrinkage

                 Comparisons significant at the 0.05 level are indicated by ***.
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                                  Difference
              time                 Between      Simultaneous 95%
           Comparison               Means     Confidence Limits

      72          - 0.6666666667   0.080611    0.065931  0.095292  ***
      72          - 0.67           0.087833    0.073153  0.102514  ***
      72          - 0.5            0.102889    0.090902  0.114876  ***
      72          - 0.15           0.139111    0.127124  0.151098  ***
      144         - 120           -0.001000   -0.012987  0.010987
      144         - 72            -0.000000   -0.011987  0.011987
      144         - 96             0.000222   -0.011764  0.012209
      144         - 48             0.003667   -0.008320  0.015653
      144         - 24             0.008444   -0.003542  0.020431
      144         - 8              0.026222    0.014236  0.038209  ***
      144         - 4              0.048611    0.036624  0.060598  ***
      144         - 3              0.051333    0.039347  0.063320  ***
      144         - 2.5            0.052833    0.040847  0.064820  ***
      144         - 2              0.054278    0.042291  0.066264  ***
      144         - 1.5            0.056000    0.044013  0.067987  ***
      144         - 1              0.063106    0.051119  0.075092  ***
      144         - 0.8333333333   0.067056    0.052375  0.081736  ***
      144         - 0.83           0.074722    0.060042  0.089403  ***
      144         - 0.6666666667   0.080611    0.065931  0.095292  ***
      144         - 0.67           0.087833    0.073153  0.102514  ***
      144         - 0.5            0.102889    0.090902  0.114876  ***
      144         - 0.15           0.139111    0.127124  0.151098  ***
      96          - 120           -0.001222   -0.013209  0.010764
      96          - 72            -0.000222   -0.012209  0.011764
      96          - 144           -0.000222   -0.012209  0.011764
      96          - 48             0.003444   -0.008542  0.015431
      96          - 24             0.008222   -0.003764  0.020209
      96          - 8              0.026000    0.014013  0.037987  ***
      96          - 4              0.048389    0.036402  0.060376  ***
      96          - 3              0.051111    0.039124  0.063098  ***
      96          - 2.5            0.052611    0.040624  0.064598  ***
      96          - 2              0.054056    0.042069  0.066042  ***
      96          - 1.5            0.055778    0.043791  0.067764  ***
      96          - 1              0.062883    0.050897  0.074870  ***
      96          - 0.8333333333   0.066833    0.052153  0.081514  ***
      96          - 0.83           0.074500    0.059819  0.089181  ***
      96          - 0.6666666667   0.080389    0.065708  0.095069  ***
      96          - 0.67           0.087611    0.072931  0.102292  ***
      96          - 0.5            0.102667    0.090680  0.114653  ***
      96          - 0.15           0.138889    0.126902  0.150876  ***
      48          - 120           -0.004667   -0.016653  0.007320

                               The SAS System        12:56 Saturday, May 26,
2001  15

                             The ANOVA Procedure

              Tukey's Studentized Range (HSD) Test for shrinkage

       Comparisons significant at the 0.05 level are indicated by ***.

                                  Difference
              time                 Between      Simultaneous 95%
           Comparison               Means     Confidence Limits

      48          - 72            -0.003667   -0.015653  0.008320
      48          - 144           -0.003667   -0.015653  0.008320
      48          - 96            -0.003444   -0.015431  0.008542
      48          - 24             0.004778   -0.007209  0.016764
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48          - 8               0.022556      0.010569  0.034542  ***
48          - 4               0.044944      0.032958  0.056931  ***
48          - 3               0.047667      0.035680  0.059653  ***
48          - 2.5             0.049167      0.037180  0.061153  ***
48          - 2               0.050611      0.038624  0.062598  ***
48          - 1.5             0.052333      0.040347  0.064320  ***
48          - 1               0.059439      0.047452  0.071426  ***
48          - 0.8333333333    0.063389      0.048708  0.078069  ***
48          - 0.83            0.071056      0.056375  0.085736  ***
48          - 0.6666666667    0.076944      0.062264  0.091625  ***
48          - 0.67            0.084167      0.069486  0.098847  ***
48          - 0.5             0.099222      0.087236  0.111209  ***
48          - 0.15            0.135444      0.123458  0.147431  ***
24          - 120            -0.009444     -0.021431  0.002542
24          - 72             -0.008444     -0.020431  0.003542
24          - 144            -0.008444     -0.020431  0.003542
24          - 96             -0.008222     -0.020209  0.003764
24          - 48             -0.004778     -0.016764  0.007209
24          - 8               0.017778      0.005791  0.029764  ***
24          - 4               0.040167      0.028180  0.052153  ***
24          - 3               0.042889      0.030902  0.054876  ***
24          - 2.5             0.044389      0.032402  0.056376  ***
24          - 2               0.045833      0.033847  0.057820  ***
24          - 1.5             0.047556      0.035569  0.059542  ***
24          - 1               0.054661      0.042674  0.066648  ***
24          - 0.8333333333    0.058611      0.043931  0.073292  ***
24          - 0.83            0.066278      0.051597  0.080958  ***
24          - 0.6666666667    0.072167      0.057486  0.086847  ***
24          - 0.67            0.079389      0.064708  0.094069  ***
24          - 0.5             0.094444      0.082458  0.106431  ***
24          - 0.15            0.130667      0.118680  0.142653  ***
8           - 120            -0.027222     -0.039209 -0.015236  ***
8           - 72             -0.026222     -0.038209 -0.014236  ***
8           - 144            -0.026222     -0.038209 -0.014236  ***
8           - 96             -0.026000     -0.037987 -0.014013  ***
8           - 48             -0.022556     -0.034542 -0.010569  ***
8           - 24             -0.017778     -0.029764 -0.005791  ***

                                The SAS System         12:56 Saturday, May 26,
2001  16

                             The ANOVA Procedure

                Tukey's Studentized Range (HSD) Test for shrinkage

         Comparisons significant at the 0.05 level are indicated by ***.


                              Difference
                 time          Between      Simultaneous 95%
              Comparison        Means      Confidence Limits

8           - 4               0.022389      0.010402  0.034376  ***
8           - 3               0.025111      0.013124  0.037098  ***
8           - 2.5             0.026611      0.014624  0.038598  ***
8           - 2               0.028056      0.016069  0.040042  ***
8           - 1.5             0.029778      0.017791  0.041764  ***
8           - 1               0.036883      0.024897  0.048870  ***
8           - 0.8333333333    0.040833      0.026153  0.055514  ***
8           - 0.83            0.048500      0.033819  0.063181  ***
8           - 0.6666666667    0.054389      0.039708  0.069069  ***
8           - 0.67            0.061611      0.046931  0.076292  ***
8           - 0.5             0.076667      0.064680  0.088653  ***
8           - 0.15            0.112889      0.100902  0.124876  ***
4           - 120            -0.049611     -0.061598 -0.037624  ***
4           - 72             -0.048611     -0.060598 -0.036624  ***
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             4            - 144             -0.048611    -0.060598 -0.036624  ***
             4            - 96              -0.048389    -0.060376 -0.036402  ***
             4            - 48              -0.044944    -0.056931 -0.032958  ***
             4            - 24              -0.040167    -0.052153 -0.028180  ***
             4            - 8               -0.022389    -0.034376 -0.010402  ***
             4            - 3                0.002722    -0.009264  0.014709
             4            - 2.5              0.004222    -0.007764  0.016209
             4            - 2                0.005667    -0.006320  0.017653
             4            - 1.5              0.007389    -0.004598  0.019376
             4            - 1                0.014494     0.002508  0.026481  ***
             4            - 0.8333333333     0.018444     0.003764  0.033125  ***
             4            - 0.83             0.026111     0.011431  0.040792  ***
             4            - 0.6666666667     0.032000     0.017319  0.046681  ***
             4            - 0.67             0.039222     0.024542  0.053903  ***
             4            - 0.5              0.054278     0.042291  0.066264  ***
             4            - 0.15             0.090500     0.078513  0.102487  ***
             3            - 120             -0.052333    -0.064320 -0.040347  ***
             3            - 72              -0.051333    -0.063320 -0.039347  ***
             3            - 144             -0.051333    -0.063320 -0.039347  ***
             3            - 96              -0.051111    -0.063098 -0.039124  ***
             3            - 48              -0.047667    -0.059653 -0.035680  ***
             3            - 24              -0.042889    -0.054876 -0.030902  ***
             3            - 8               -0.025111    -0.037098 -0.013124  ***
             3            - 4               -0.002722    -0.014709  0.009264
             3            - 2.5              0.001500    -0.010487  0.013487
             3            - 2                0.002944    -0.009042  0.014931
             3            - 1.5              0.004667    -0.007320  0.016653

                                        The SAS System         12:56 Saturday, May 26,
2001  17

                                     The ANOVA Procedure

                       Tukey's Studentized Range (HSD) Test for shrinkage

                Comparisons significant at the 0.05 level are indicated by ***.


                                           Difference
                        time                  Between     Simultaneous 95%
                     Comparison                 Means    Confidence Limits

             3            - 1                0.011772    -0.000214  0.023759
             3            - 0.8333333333     0.015722     0.001042  0.030403  ***
             3            - 0.83             0.023389     0.008708  0.038069  ***
             3            - 0.6666666667     0.029278     0.014597  0.043958  ***
             3            - 0.67             0.036500     0.021819  0.051181  ***
             3            - 0.5              0.051556     0.039569  0.063542  ***
             3            - 0.15             0.087778     0.075791  0.099764  ***
             2.5          - 120             -0.053833    -0.065820 -0.041847  ***
             2.5          - 72              -0.052833    -0.064820 -0.040847  ***
             2.5          - 144             -0.052833    -0.064820 -0.040847  ***
             2.5          - 96              -0.052611    -0.064598 -0.040624  ***
             2.5          - 48              -0.049167    -0.061153 -0.037180  ***
             2.5          - 24              -0.044389    -0.056376 -0.032402  ***
             2.5          - 8               -0.026611    -0.038598 -0.014624  ***
             2.5          - 4               -0.004222    -0.016209  0.007764
             2.5          - 3               -0.001500    -0.013487  0.010487
             2.5          - 2                0.001444    -0.010542  0.013431
             2.5          - 1.5              0.003167    -0.008820  0.015153
             2.5          - 1                0.010272    -0.001714  0.022259
             2.5          - 0.8333333333     0.014222    -0.000458  0.028903
             2.5          - 0.83             0.021889     0.007208  0.036569  ***
             2.5          - 0.6666666667     0.027778     0.013097  0.042458  ***
             2.5          - 0.67             0.035000     0.020319  0.049681  ***
             2.5          - 0.5              0.050056     0.038069  0.062042  ***
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           2.5          - 0.15             0.086278      0.074291  0.098264  ***
           2            - 120             -0.055278     -0.067264 -0.043291  ***
           2            - 72              -0.054278     -0.066264 -0.042291  ***
           2            - 144             -0.054278     -0.066264 -0.042291  ***
           2            - 96              -0.054056     -0.066042 -0.042069  ***
           2            - 48              -0.050611     -0.062598 -0.038624  ***
           2            - 24              -0.045833     -0.057820 -0.033847  ***
           2            - 8               -0.028056     -0.040042 -0.016069  ***
           2            - 4               -0.005667     -0.017653  0.006320
           2            - 3               -0.002944     -0.014931  0.009042
           2            - 2.5             -0.001444     -0.013431  0.010542
           2            - 1.5              0.001722     -0.010264  0.013709
           2            - 1                0.008828     -0.003159  0.020814
           2            - 0.8333333333     0.012778     -0.001903  0.027458
           2            - 0.83             0.020444      0.005764  0.035125  ***
           2            - 0.6666666667     0.026333      0.011653  0.041014  ***
           2            - 0.67             0.033556      0.018875  0.048236  ***

                                         The SAS System        12:56 Saturday, May 26,
2001  18

                                      The ANOVA Procedure

                         Tukey's Studentized Range (HSD) Test for shrinkage

                    Comparisons significant at the 0.05 level are indicated by ***.


                                         Difference
                       time               Between      Simultaneous 95%
                    Comparison             Means      Confidence Limits

           2            - 0.5              0.048611      0.036624  0.060598  ***
           2            - 0.15             0.084833      0.072847  0.096820  ***
           1.5          - 120             -0.057000     -0.068987 -0.045013  ***
           1.5          - 72              -0.056000     -0.067987 -0.044013  ***
           1.5          - 144             -0.056000     -0.067987 -0.044013  ***
           1.5          - 96              -0.055778     -0.067764 -0.043791  ***
           1.5          - 48              -0.052333     -0.064320 -0.040347  ***
           1.5          - 24              -0.047556     -0.059542 -0.035569  ***
           1.5          - 8               -0.029778     -0.041764 -0.017791  ***
           1.5          - 4               -0.007389     -0.019376  0.004598
           1.5          - 3               -0.004667     -0.016653  0.007320
           1.5          - 2.5             -0.003167     -0.015153  0.008820
           1.5          - 2               -0.001722     -0.013709  0.010264
           1.5          - 1                0.007106     -0.004881  0.019092
           1.5          - 0.8333333333     0.011056     -0.003625  0.025736
           1.5          - 0.83             0.018722      0.004042  0.033403  ***
           1.5          - 0.6666666667     0.024611      0.009931  0.039292  ***
           1.5          - 0.67             0.031833      0.017153  0.046514  ***
           1.5          - 0.5              0.046889      0.034902  0.058876  ***
           1.5          - 0.15             0.083111      0.071124  0.095098  ***
           1            - 120             -0.064106     -0.076092 -0.052119  ***
           1            - 72              -0.063106     -0.075092 -0.051119  ***
           1            - 144             -0.063106     -0.075092 -0.051119  ***
           1            - 96              -0.062883     -0.074870 -0.050897  ***
           1            - 48              -0.059439     -0.071426 -0.047452  ***
           1            - 24              -0.054661     -0.066648 -0.042674  ***
           1            - 8               -0.036883     -0.048870 -0.024897  ***
           1            - 4               -0.014494     -0.026481 -0.002508  ***
           1            - 3               -0.011772     -0.023759  0.000214
           1            - 2.5             -0.010272     -0.022259  0.001714
           1            - 2               -0.008828     -0.020814  0.003159
           1            - 1.5             -0.007106     -0.019092  0.004881
           1            - 0.8333333333     0.003950     -0.010731  0.018631
           1            - 0.83             0.011617     -0.003064  0.026297
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            1            - 0.6666666667       0.017506       0.002825  0.032186  ***
            1            - 0.67               0.024728       0.010047  0.039408  ***
            1            - 0.5                0.039783       0.027797  0.051770  ***
            1            - 0.15               0.076006       0.064019  0.087992  ***
            0.8333333333 - 120               -0.068056      -0.082736 -0.053375  ***
            0.8333333333 - 72                -0.067056      -0.081736 -0.052375  ***
            0.8333333333 - 144               -0.067056      -0.081736 -0.052375  ***

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                                   The ANOVA Procedure

                        Tukey's Studentized Range (HSD) Test for shrinkage

                    Comparisons significant at the 0.05 level are indicated by ***.


                                             Difference
                     time                     Between        Simultaneous 95%
                  Comparison                    Means        Confidence Limits

            0.8333333333 - 96                -0.066833      -0.081514 -0.052153  ***
            0.8333333333 - 48                -0.063389      -0.078069 -0.048708  ***
            0.8333333333 - 24                -0.058611      -0.073292 -0.043931  ***
            0.8333333333 - 8                 -0.040833      -0.055514 -0.026153  ***
            0.8333333333 - 4                 -0.018444      -0.033125 -0.003764  ***
            0.8333333333 - 3                 -0.015722      -0.030403 -0.001042  ***
            0.8333333333 - 2.5               -0.014222      -0.028903  0.000458
            0.8333333333 - 2                 -0.012778      -0.027458  0.001903
            0.8333333333 - 1.5               -0.011056      -0.025736  0.003625
            0.8333333333 - 1                 -0.003950      -0.018631  0.010731
            0.8333333333 - 0.83               0.007667      -0.009285  0.024618
            0.8333333333 - 0.6666666667       0.013556      -0.003396  0.030507
            0.8333333333 - 0.67               0.020778       0.003826  0.037729  ***
            0.8333333333 - 0.5                0.035833       0.021153  0.050514  ***
            0.8333333333 - 0.15               0.072056       0.057375  0.086736  ***
            0.83         - 120               -0.075722      -0.090403 -0.061042  ***
            0.83         - 72                -0.074722      -0.089403 -0.060042  ***
            0.83         - 144               -0.074722      -0.089403 -0.060042  ***
            0.83         - 96                -0.074500      -0.089181 -0.059819  ***
            0.83         - 48                -0.071056      -0.085736 -0.056375  ***
            0.83         - 24                -0.066278      -0.080958 -0.051597  ***
            0.83         - 8                 -0.048500      -0.063181 -0.033819  ***
            0.83         - 4                 -0.026111      -0.040792 -0.011431  ***
            0.83         - 3                 -0.023389      -0.038069 -0.008708  ***
            0.83         - 2.5               -0.021889      -0.036569 -0.007208  ***
            0.83         - 2                 -0.020444      -0.035125 -0.005764  ***
            0.83         - 1.5               -0.018722      -0.033403 -0.004042  ***
            0.83         - 1                 -0.011617      -0.026297  0.003064
            0.83         - 0.8333333333      -0.007667      -0.024618  0.009285
            0.83         - 0.6666666667       0.005889      -0.011063  0.022841
            0.83         - 0.67               0.013111      -0.003841  0.030063
            0.83         - 0.5                0.028167       0.013486  0.042847  ***
            0.83         - 0.15               0.064389       0.049708  0.079069  ***
            0.6666666667 - 120               -0.081611      -0.096292 -0.066931  ***
            0.6666666667 - 72                -0.080611      -0.095292 -0.065931  ***
            0.6666666667 - 144               -0.080611      -0.095292 -0.065931  ***
            0.6666666667 - 96                -0.080389      -0.095069 -0.065708  ***
            0.6666666667 - 48                -0.076944      -0.091625 -0.062264  ***
            0.6666666667 - 24                -0.072167      -0.086847 -0.057486  ***
            0.6666666667 - 8                 -0.054389      -0.069069 -0.039708  ***
            0.6666666667 - 4                 -0.032000      -0.046681 -0.017319  ***

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2001  20
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                          The ANOVA Procedure

            Tukey's Studentized Range (HSD) Test for shrinkage

       Comparisons significant at the 0.05 level are indicated by ***.

                                    Difference
              time                   Between      Simultanecus 95%
           Comparison                 Means       Confidence Limits

0.6666666667 - 3                   -0.029278    -0.043958 -0.014597  ***
0.6666666667 - 2.5                 -0.027778    -0.042458 -0.013097  ***
0.6666666667 - 2                   -0.026333    -0.041014 -0.011653  ***
0.6666666667 - 1.5                 -0.024611    -0.039292 -0.009931  ***
0.6666666667 - 1                   -0.017506    -0.032186 -0.002825  ***
0.6666666667 - 0.8333333333        -0.013556    -0.030507  0.003396
0.6666666667 - 0.83                -0.005889    -0.022841  0.011063
0.6666666667 - 0.67                 0.007222    -0.009729  0.024174
0.6666666667 - 0.5                  0.022278     0.007597  0.036958  ***
0.6666666667 - 0.15                 0.058500     0.043819  0.073181  ***
0.67         - 120                 -0.088833    -0.103514 -0.074153  ***
0.67         - 72                  -0.087833    -0.102514 -0.073153  ***
0.67         - 144                 -0.087833    -0.102514 -0.073153  ***
0.67         - 96                  -0.087611    -0.102292 -0.072931  ***
0.67         - 48                  -0.084167    -0.098847 -0.069486  ***
0.67         - 24                  -0.079389    -0.094069 -0.064708  ***
0.67         - 8                   -0.061611    -0.076292 -0.046931  ***
0.67         - 4                   -0.039222    -0.053903 -0.024542  ***
0.67         - 3                   -0.036500    -0.051181 -0.021819  ***
0.67         - 2.5                 -0.035000    -0.049681 -0.020319  ***
0.67         - 2                   -0.033556    -0.048236 -0.018875  ***
0.67         - 1.5                 -0.031833    -0.046514 -0.017153  ***
0.67         - 1                   -0.024728    -0.039408 -0.010047  ***
0.67         - 0.8333333333        -0.020778    -0.037729 -0.003826  ***
0.67         - 0.83                -0.013111    -0.030063  0.003841
0.67         - 0.6666666667        -0.007222    -0.024174  0.009729
0.67         - 0.5                  0.015056     0.000375  0.029736  ***
0.67         - 0.15                 0.051278     0.036597  0.065958  ***
0.5          - 120                 -0.103889    -0.115876 -0.091902  ***
0.5          - 72                  -0.102889    -0.114876 -0.090902  ***
0.5          - 144                 -0.102889    -0.114876 -0.090902  ***
0.5          - 96                  -0.102667    -0.114653 -0.090680  ***
0.5          - 48                  -0.099222    -0.111209 -0.087236  ***
0.5          - 24                  -0.094444    -0.106431 -0.082458  ***
0.5          - 8                   -0.076667    -0.088653 -0.064680  ***
0.5          - 4                   -0.054278    -0.066264 -0.042291  ***
0.5          - 3                   -0.051556    -0.063542 -0.039569  ***
0.5          - 2.5                 -0.050056    -0.062042 -0.038069  ***
0.5          - 2                   -0.048611    -0.060598 -0.036624  ***
0.5          - 1.5                 -0.046889    -0.058876 -0.034902  ***
0.5          - 1                   -0.039783    -0.051770 -0.027797  ***

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2001  21

                          The ANOVA Procedure

            Tukey's Studentized Range (HSD) Test for shrinkage

       Comparisons significant at the 0.05 level are indicated by ***.

                                    Difference
              time                   Between      Simultaneous 95%
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                 Comparison                 Means     Confidence Limits

        0.5          - 0.8333333333     -0.035833    -0.050514 -0.021153  ***
        0.5          - 0.83             -0.028167    -0.042847 -0.013486  ***
        0.5          - 0.6666666667     -0.022278    -0.036958 -0.007597  ***
        0.5          - 0.67             -0.015056    -0.029736 -0.000375  ***
        0.5          - 0.15              0.036222     0.024236  0.048209  ***
        0.15         - 120              -0.140111    -0.152098 -0.128124  ***
        0.15         - 72               -0.139111    -0.151098 -0.127124  ***
        0.15         - 144              -0.139111    -0.151098 -0.127124  ***
        0.15         - 96               -0.138889    -0.150876 -0.126902  ***
        0.15         - 48               -0.135444    -0.147431 -0.123458  ***
        0.15         - 24               -0.130667    -0.142653 -0.118680  ***
        0.15         - 8                -0.112889    -0.124876 -0.100902  ***
        0.15         - 4                -0.090500    -0.102487 -0.078513  ***
        0.15         - 3                -0.087778    -0.099764 -0.075791  ***
        0.15         - 2.5              -0.086278    -0.098264 -0.074291  ***
        0.15         - 2                -0.084833    -0.096820 -0.072847  ***
        0.15         - 1.5              -0.083111    -0.095098 -0.071124  ***
        0.15         - 1                -0.076006    -0.087992 -0.064019  ***
        0.15         - 0.8333333333     -0.072056    -0.086736 -0.057375  ***
        0.15         - 0.83             -0.064389    -0.079069 -0.049708  ***
        0.15         - 0.6666666667     -0.058500    -0.073181 -0.043819  ***
        0.15         - 0.67             -0.051278    -0.065958 -0.036597  ***
        0.15         - 0.5              -0.036222    -0.048209 -0.024236  ***

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2001  22

                                 The ANOVA Procedure

                              t Tests (LSD) for shrinkage

   NOTE: This test controls the Type I comparisonwise error rate, not the experimentwise
error
                                       rate.

                              Alpha                        0.05
                              Error Degrees of Freedom      134
                              Error Mean Square          0.0001
                              Critical Value of t       1.97783

                 Comparisons significant at the 0.05 level are indicated by ***.

                                  Difference
                       Ag           Between
                   Comparison        Means    95% Confidence Limits

                   0     - 50      0.1085632    0.1061594  0.1109670  ***
                   50    - 0      -0.1085632   -0.1109670 -0.1061594  ***
                                    The SAS System        12:56 Saturday, May 26,
2001  23

                                 The ANOVA Procedure

                      Tukey's Studentized Range (HSD) Test for shrinkage

                 NOTE: This test controls the Type I experimentwise error rate.

                           Alpha                                  0.05
                           Error Degrees of Freedom                134
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                      Error Mean Square                     0.0001
                      Critical Value of Studentized Range  2.79707


            Comparisons significant at the 0.05 level are indicated by ***.


                               Difference
                  Ag            Between      Simultaneous 95%
              Comparison          Means      Confidence Limits

              0     - 50       0.1085632     0.1061594  0.1109670   ***
              50    - 0       -0.1085632    -0.1109670 -0.1061594   ***

                                     The SAS System         12:56 Saturday, May 26,
2001  24

                                  The ANOVA Procedure

                               t Tests (LSD) for shrinkage

  NOTE: This test controls the Type I comparisonwise error rate, not the experimentwise
error
                                         rate.


                           Alpha                         0.05
                           Error Degrees of Freedom       134
                           Error Mean Square           0.0001
                           Critical Value of t        1.97783


            Comparisons significant at the 0.05 level are indicated by ***.


                               Difference
                  FG            Between
              Comparison          Means    95% Confidence Limits

              0     - 1        0.0840985     0.0816947  0.0865023   ***
              1     - 0       -0.0840985    -0.0865023 -0.0816947   ***

                                     The SAS System         12:56 Saturday, May 26,
2001  25

                                  The ANOVA Procedure

                    Tukey's Studentized Range (HSD) Test for shrinkage

             NOTE: This test controls the Type I experimentwise error rate.


                      Alpha                                   0.05
                      Error Degrees of Freedom                 134
                      Error Mean Square                     0.0001
                      Critical Value of Studentized Range  2.79707


            Comparisons significant at the 0.05 level are indicated by ***.


                               Difference
                  FG            Between      Simultaneous 95%
              Comparison          Means      Confidence Limits

              0     - 1        0.0840985     0.0816947  0.0865023   ***
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                    1     - 0       -0.0840985    -0.0865023 -0.0816947  ***
                                         The SAS System        12:56 Saturday, May 26,
2001  26

                                       The ANOVA Procedure

                                    t Tests (LSD) for shrinkage

  NOTE: This test controls the Type I comparisonwise error rate, not the experimentwise
error
                                              rate.


                         Alpha                            0.05
                         Error Degrees of Freedom          134
                         Error Mean Square              0.0001
                         Critical Value of t           1.97783
                         Least Significant Difference   0.0028


                Comparisons significant at the 0.05 level are indicated by ***.


                                           Difference
                       Material              Between       95% Confidence
                      Comparison               Means           Limits

               Sulfur       - Sulfur_3%DCP    0.068216     0.065440  0.070991  ***
               Sulfur       - Sulfur_5%DCP    0.150205     0.147429  0.152981  ***
               Sulfur_3%DCP - Sulfur         -0.068216    -0.070991 -0.065440  ***
               Sulfur_3%DCP - Sulfur_5%DCP    0.081989     0.079214  0.084765  ***
               Sulfur_5%DCP - Sulfur         -0.150205    -0.152981 -0.147429  ***
               Sulfur_5%DCP - Sulfur_3%DCP   -0.081989    -0.084765 -0.079214  ***
                                         The SAS System        12:56 Saturday, May 26,
2001  27

                                       The ANOVA Procedure

                         Tukey's Studentized Range (HSD) Test for shrinkage

                 NOTE: This test controls the Type I experimentwise error rate.


                      Alpha                                  0.05
                      Error Degrees of Freedom                134
                      Error Mean Square                    0.0001
                      Critical Value of Studentized Range 3.35179
                      Minimum Significant Difference       0.0033


                Comparisons significant at the 0.05 level are indicated by ***.


                                           Difference
                       Material              Between      Simultaneous 95%
                      Comparison               Means     Confidence Limits

               Sulfur       - Sulfur_3%DCP    0.068216     0.064890  0.071542  ***
               Sulfur       - Sulfur_5%DCP    0.150205     0.146879  0.153531  ***
               Sulfur_3%DCP - Sulfur         -0.068216    -0.071542 -0.064890  ***
               Sulfur_3%DCP - Sulfur_5%DCP    0.081989     0.078663  0.085315  ***
               Sulfur_5%DCP - Sulfur         -0.150205    -0.153531 -0.146879  ***
               Sulfur_5%DCP - Sulfur_3%DCP   -0.081989    -0.085315 -0.078663  ***
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