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Sulfapave: Densifiable Sulfur Concrete Materials for Corrosive Environments

By W. C. McBee, T. A. Sullivan, and B. W. Jong



UNITED STATES DEPARTMENT OF THE INTERIOR

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UNIT OF MEASURE ABBREVIATIONS USED IN THIS REPORT

°C	degree Celsius	lb	pound
cp	centipoise	min	minute
°C/min	degree Celsius per minute	mm	millimeter
c/min	cycle per minute	pct	percent
ft	foot	psi	pound per square inch
h	hour	t	metric ton
in	inch		

SULFAPAVE: DENSIFIABLE SULFUR CONCRETE MATERIALS FOR CORROSIVE ENVIRONMENTS

By W. C. McBee,¹ T. A. Sullivan,² and B. W. Jong³

ABSTRACT

Technology for the preparation of densifiable sulfur concrete (sulfapave) materials has been devised by the Bureau of Mines. The materials are intended as new construction materials for use in corrosive areas where conventional materials fail. A series of modified sulfur cements were developed, which can be used in combination with dense-graded aggregates to prepare sulfapave materials with excellent corrosion-resistant properties. The materials can be mixed, laid, and compacted into place with conventional hot-mix asphalt paving equipment. Mixture designs utilizing 5- to 30-pct-modified sulfur cements and dense-graded aggregates gave material properties ranging from rigid to flexible. Laboratory corrosion tests in sulfuric acid showed no material loss or deterioration of physical properties over a 1-year test period in concentrations ranging from 0 to 60 pct at ambient temperatures. Preliminary results on field testing the materials for durability to weathering, corrosion, and traffic have been excellent, with no evidence of failure in the materials.

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INTRODUCTION

The recent incentive to use sulfur in construction materials began in the early 1970's. Increasing amounts of secondary sulfur were becoming available through their recovery from new sour-gas wells, the refining of sulfur-containing crude oil, and potentially, from environmental sources such as metal smelters, coal-burning powerplants, and synthetic fuels from coal. Forecasts were being made for large increases in secondary sulfur production through the year 2000 (1).⁴

Because the market for sulfur is inelastic, large amounts of secondary sulfur coming on the market would cause storage and economic problems. The U.S. Department of the Interior initiated a sulfur utilization program in 1972 to devise new uses for this readily available and valuable raw material. The most promising use of large amounts of sulfur was in construction materials. With the developing energy crisis, the conservation of energy-intensive materials such as asphalt and portland cement by utilizing sulfur in paving and construction materials appeared feasible from both an economic basis and practical basis, as demonstrated by the performance of some of the newly developed sulfur construction materials.

Bureau research on sulfur utilization in construction materials in the last 11 years has shown many areas where sulfur

may be substituted for more energy-intensive materials. Sulfur materials have been produced that have properties equal to or, in most cases, superior to those of materials currently used. Some of these products are sulfur-extended asphalt paving, sulfur-sand-asphalt paving, recycled spent asphalt paving with sulfur, sulfur composite materials, modified sulfur cements, and corrosion-resistant direct-cast conventional sulfur concretes (2-22). In addition to the Bureau's work in these areas, increasing numbers of other research groups are engaging in the development and utilization of sulfur in construction materials (23-39).

This report presents the development of technology for the preparation of densifiable sulfur concretes. Modified sulfur cements were developed for use with suitable aggregates to prepare sulfur concretes that can be produced in commercial asphalt paving plants and laid and compacted with conventional asphalt paving equipment. Sulfur concrete materials were devised for use in areas where acid and salt corrosive solutions destroy construction materials currently used. These include such uses as acid loading docks, floors, roadways, bridge decking, pipe, culverts, and as impervious liners for hazardous liquid storage ponds in metallurgical, chemical, and fertilizer plants.

MODIFIED SULFUR CEMENTS

Laboratory-scale development of modified sulfur cements for use in the preparation of densifiable sulfur concrete materials has been reported (11-12, 15-17). The cements are prepared by reacting a mixture of molten sulfur, dicyclopentadiene (DCPD), and oligomers of cyclopentadiene (16), primarily the trimer through the pentamer, hereafter referred to as "oligomer." The reaction products

are stabilized linear polymeric polysulfides. By varying the proportions of DCPD and oligomer reacting with the sulfur, cements of different physical properties may be prepared which can be used with suitable aggregates to prepare densifiable-sulfur concrete materials ranging from rigid concretes to flexible (viscoelastic) concretes.

This report presents the results obtained on scaling up the preparation of the cements from 2.2-lb batches to 1,300-lb batches and the use of the cements to

⁴Underlined numbers in parentheses refer to items in the list of references at the end of this report.

prepare sulfur concrete. Scale-up to larger batches was done in two types of reactors. An electrically heated reactor (fig. 1) was used to prepare 500-lb batches of the sulfur cements. A steam-heated reactor (fig. 2) was used in preparing 1,300-lb sulfur cement batches. Both were sealed reactors that allowed the more volatile components to reflux back into the stirred reaction mixture to ensure complete reaction of the components. The steam-heated reactor was superior to the electrically heated reactor because of faster melting of solid flake sulfur, better control of the reactor temperature, and safer use with potential flammable materials. Reaction temperatures ranged from 120° to 146° C, and most reactions were held for 24 h. Normally, the reaction was essentially complete within 12 h after adding the organic materials into the liquid sulfur in the reactor.

PREPARATION

In scaling-up the preparation of various types of modified sulfur cements from laboratory to 1,350-lb batches, it was necessary to establish the optimum proportions of the chemical modifiers, DCPD, and oligomer, to react with the sulfur. Addition of an excess of DCPD resulted in an uncontrollable exothermic reaction; an excess of oligomer resulted in incomplete reaction with sulfur. For example, a cement suitable for preparing rigid densifiable sulfur concrete is made by reacting sulfur with 5 pct of a 50 pct DCPD, 50 pct oligomer mixture;⁵ however, in preparing a cement with viscoelastic properties similar to asphaltic cement, the sulfur is reacted with up to 30 pct of a 15 pct DCPD, 85 pct oligomer mixture

⁵All percentage values used in this paper are weight percents.

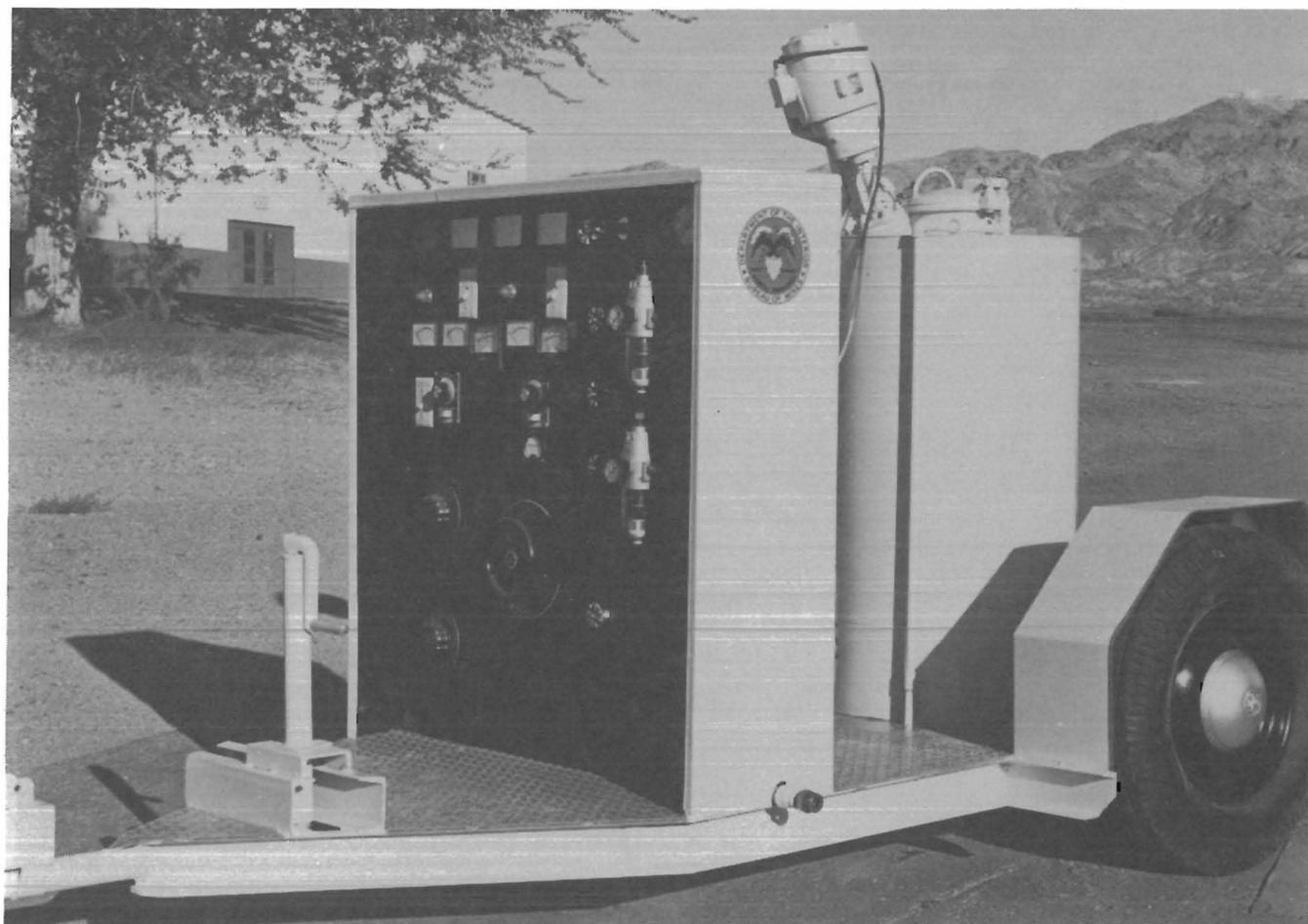


FIGURE 1. - Electrically heated 500-lb sulfur cement reactor.

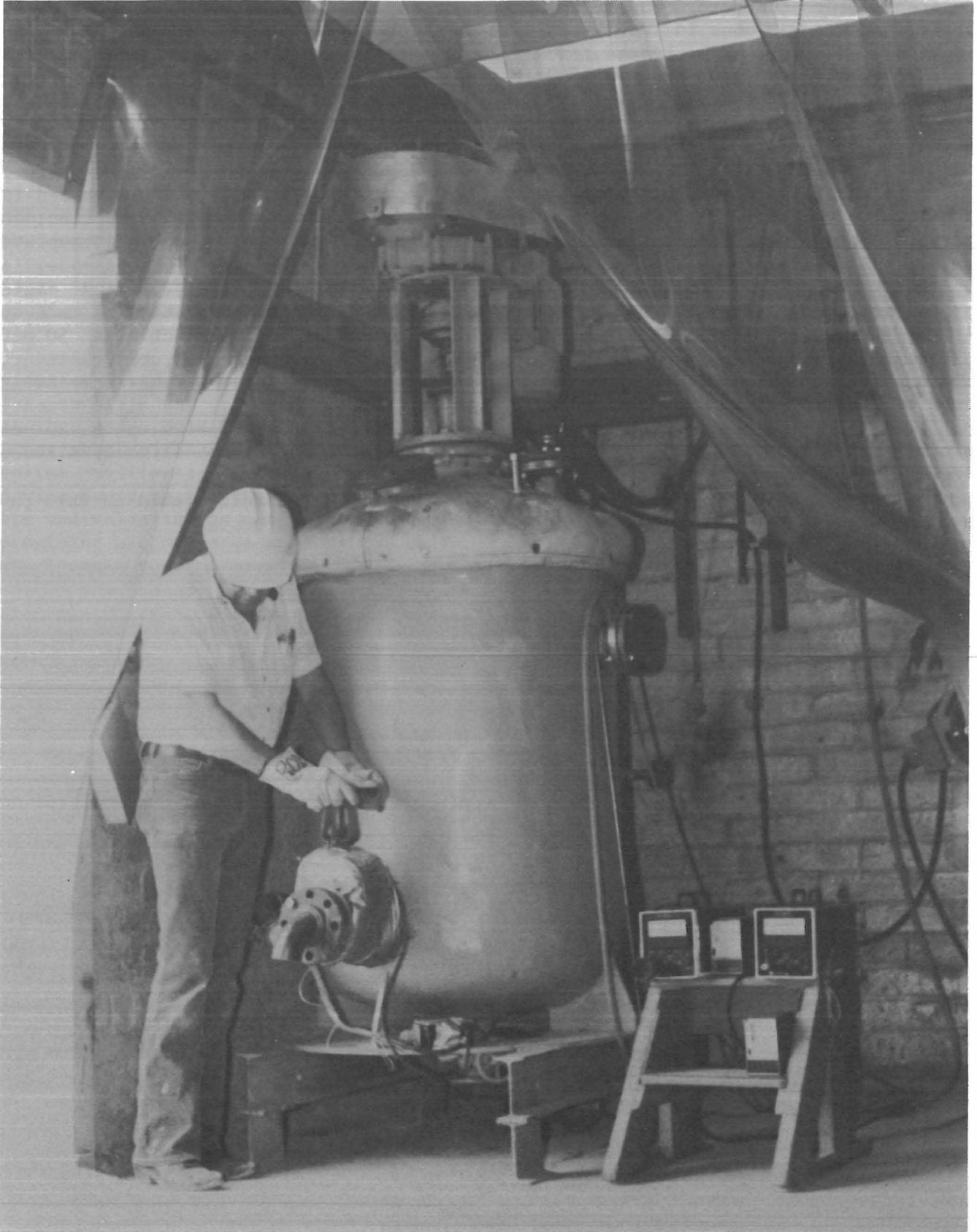


FIGURE 2. - Steam-heated 1-t sulfur cement reactor.

The optimum modifier proportions, using 97-pct-grade DCPD and commercial-grade oligomer for preparing various types of sulfur cement were as follows, in percent:

Cement		Modifier composition	
Sulfur	Modifier	DCPD	Oligomer
95	5	50	50
90	10	20	80
85	15	20	80
80	20	15	85
75	25	15	85
70	30	15	85

When using the large-scale steam-reactor, the chemical modifiers were charged into stirred molten sulfur at 140° C, and the reactor was closed and held at 140° C for 24 h before removal and solidification of the modified cement. Using the optimum proportions of modifiers, 1,300-lb lots of various types of cement were prepared with reproducible properties for each type of cement.

PROPERTIES

Typical physical properties of the modified sulfur cements are given in table 1, along with properties of AR 4000-grade asphalt. Chemical compositions for the sulfur cements are listed in table 2. The solid cement composition of the 40-pct cement was similar to that of the 30-pct cement. This product had an excess of modifiers, as shown by the presence of liquid modifiers on solidification of the cement. The properties of the asphalt cement are typical of those used in preparing flexible, hot-mix asphaltic concrete.

TABLE 1. - Physical properties of typical modified sulfur cements and asphalt cement

Type of cement	Modifier, pct	Penetration, 0.01 mm	Softening point, °C	Sp. gr.	Viscosity at 135° C, cp
Sulfur.....	5	0	115	1.899	28
	10	0	113	1.760	35
	15	5	103	1.712	91
	20	32	103	1.649	80
	25	37	101	1.622	140
	30	45	94	1.613	169
	40	245	55	1.485	176
	Asphalt ¹	None	41	49	1.020

¹Ar 4000.

Viscosity profiles from data obtained in the preparation of typical lots of the modified cements are plotted in figure 3. Completion of the reaction between the modifiers and the sulfur is indicated when a steady-state viscosity is reached. For 5- and 10-pct-modified cements, reaction is complete in 4 to 6 h. The higher cements, 15- to 30-pct-modified, require about 16 h to complete the reaction.

Thermal expansion measurements were made in the solid state on sulfur and on modified sulfur cements. Solid samples of the cements were prepared and aged for 1 month before testing. Table 3 lists the thermal expansion coefficients of the materials. The results indicate slightly higher expansion coefficients for modified sulfur cements compared to sulfur. The 15- to 30-pct-modified cements also had lower softening points.

Differential Scanning Calorimetry (DSC) analyses were made on sulfur and modified sulfur cements. Thermograms of sulfur, 5-, 30-, and 40-pct-modified cements after 4 months aging and those of 10-, 15-, 20-, and 25-pct-modified cements after 1 month aging are shown in figure 4. The results shown indicate the phase transformation peaks, S α to S β (orthorhombic to monoclinic) and S β to S λ (monoclinic to liquid) of sulfur are superimposed on the modified cements in the 10- to 25-pct range. The extent of S α and S β crystallization decreased with increased chemical modification of the cements. The 30- and 40-pct cements showed a single phase in the solid state.

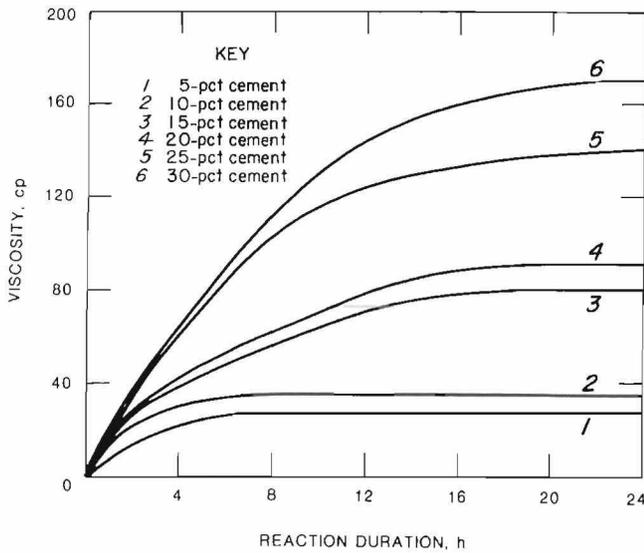


FIGURE 3. - Viscosity versus reaction time for sulfur cements.

TABLE 2. - Chemical composition of typical modified sulfur cements, percent

Sulfur cement modifier	Carbon	Hydrogen	Sulfur
5.....	4.5	0.3	95.0
10.....	7.0	.8	90.2
15.....	11.0	1.2	85.4
20.....	15.2	1.4	83.3
25.....	16.8	1.7	80.5
30.....	26.4	2.5	70.6
40.....	27.0	2.6	70.1

TABLE 3. - Thermal expansion coefficients of sulfur and sulfur cements

Modifier, pct	Thermal expansion coefficient, in/in·°C ⁻¹	Temperature range, °C
0.....	46 × 10 ⁻⁶	25-95
5.....	59 × 10 ⁻⁶	25-95
10.....	85 × 10 ⁻⁶	25-60
20.....	80 × 10 ⁻⁶	25-40
30.....	80 × 10 ⁻⁶	25-45

Thermogravimetric analysis measurements were made on sulfur and the modified cements by heating from 30° to 200° C at 10° C/min. Figure 5 illustrates the weight loss of the various cements at 140° C, the optimum temperature for preparing sulfur concretes. With the 5-pct-modified cement used to prepare rigid

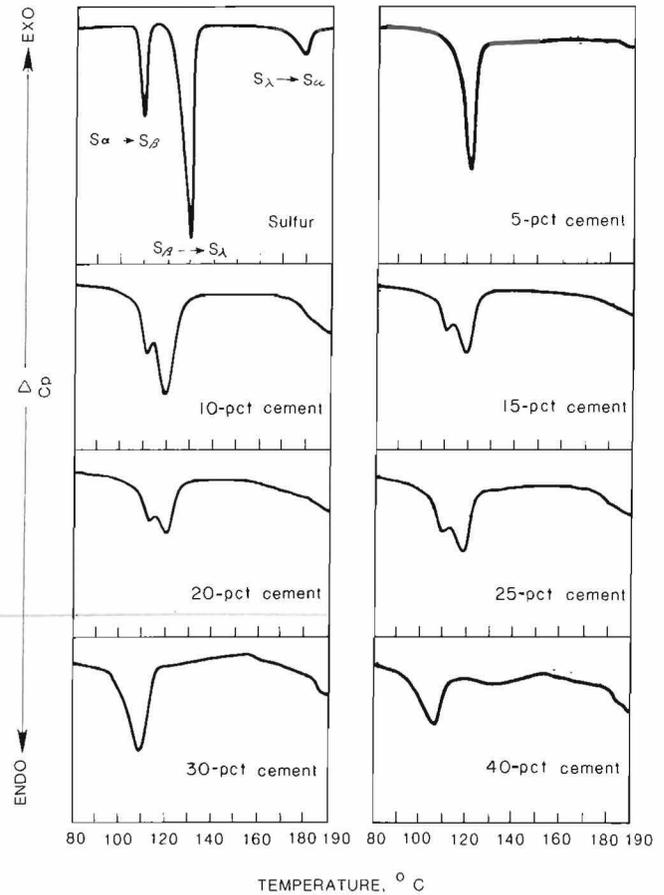


FIGURE 4. - Differential thermal analysis curves of sulfur and sulfur cements.

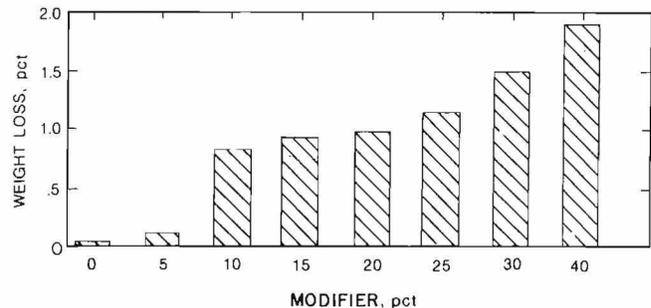


FIGURE 5. - Weight loss at 140° C on heating sulfur cements from 30° to 200° C at a rate of 10° C/min.

sulfur concrete, there is very little loss or vaporization at the mix temperature. The amount of vaporization increases with the amount of chemical modifiers in the cement. This is consistent with the results obtained on the softening points of the various cements.

SULFAPAVE

The objective of the research on sulfapave (densifiable-sulfur-concrete materials), was to make corrosion resistant, densifiable paving that could be mixed, placed, and compacted with conventional construction equipment. By using suitable modified sulfur cements and aggregates, sulfapave materials were developed that would reduce the amount of sulfur cement required for rigid direct-cast conventional sulfur concretes and could be prepared in commercially available equipment such as hot-mix asphalt plants, laid, and compacted in the same manner as flexible asphalt paving.

DEVELOPMENT

In developing sulfapave materials, dense-graded quartz aggregates, similar to those used in preparing rigid direct-cast sulfur concretes (17), were used. Mixture designs were developed to obtain paving materials that would conform to Marshall Design Specifications for high traffic density, hot-mix asphalt paving (40). Using 3/8-in dense-graded quartz aggregate and AR 4000-grade asphalt cement, the optimum binder requirement was 6 pct. With sulfapave materials prepared with 5- to 40-pct-modified sulfur cement, the optimum binder requirements were in the 9- to 11-pct range. For comparison, the properties of asphalt concrete prepared with 6 pct asphalt cement and those of sulfapave prepared with 9 pct of a 30-pct-modified sulfur cement are as follows:

<u>Property</u>	<u>Asphalt concrete</u>	<u>Sulfa-pave</u>
Marshall stability...lb..	3,660	3,220
Marshall flow...0.01 in..	10	12
Voids.....pct..	2.1	3.3
Specific gravity.....	2.354	2.385

From these values, it is apparent that sulfapave materials with initial properties similar to those of asphalt paving can be prepared.

Because of the temperature at which unreacted sulfur solidifies (118° C), sulfapave materials are more sensitive to compaction temperatures than are asphaltic concretes. The effect of compaction temperature on the stability and void contents of sulfapave material prepared with four types of sulfur cements is illustrated in figures 6 and 7. The most effective compaction temperature range for laying and compacting sulfapave prepared with 20- and 30-pct-modified cements was 120° to 145° C.

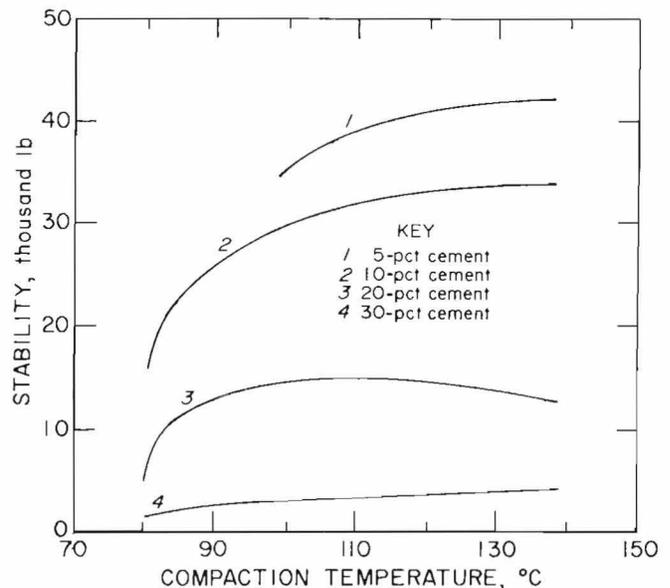


FIGURE 6. - Marshall stability values versus compaction temperature for sulfur concretes.

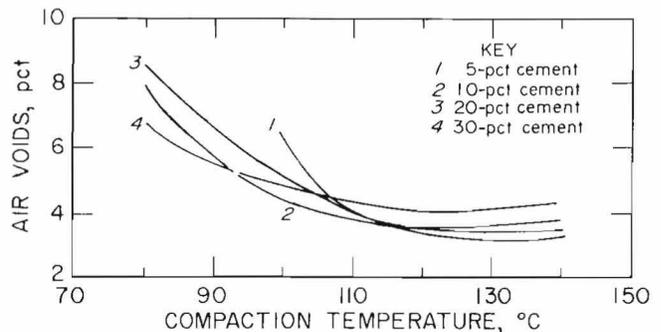


FIGURE 7. - Voids content versus compaction temperature for sulfur concretes.

In developing sulfapave products, materials were prepared with optimum binder requirements from 3/8-in quartz aggregates and with 5- to 40-pct-modified sulfur cements. Table 4 lists the properties of typical sulfapave material prepared with each type of cement. The materials were mixed at 140° C and compacted at 135° C. From the values shown in table 4 and from experience in mixing and compacting various sulfapave materials, the concretes prepared from 15- to 30-pct-modified sulfur cements were found to be best suited for use as flexible sulfur paving materials.

The resistance of sulfapave materials to acid corrosion and water damage was evaluated. Samples of sulfapave prepared with dense-graded, 3/8-in quartz aggregate and 5-, 10-, 20-, 30-, and 40-pct modified cement were tested by immersion in water and in 10-, 20-, and 60-pct-H₂SO₄ solutions for 1 yr. The samples were monitored by determining any change in weight, physical condition, or stability values on a quarterly basis. In general, no weight change or physical degradation was observed over the 1-yr period.

Figure 8 shows plots of the stability values over the test period. In general, the materials attained their maximum stability in 3 months, and the increase in stability was inversely proportional to the amount of modifier in the cement. The results indicate that the materials are not weakened by exposure to acid solutions. As indicated in the plots, samples immersed in water showed little

if any increase in stability, while those immersed in 60-pct-H₂SO₄ solutions had the highest increase in stability over the 1-yr test period.

The compressive strength of sulfapave was determined on bars prepared using a California Kneading Compactor.⁶ Compressive strength obtained on sulfapave were as follows:

Modifier, pct	Binder, pct	Compressive strength, psi
5.....	11	4,600
10.....	11	4,200
20.....	11	1,700
30.....	9	900
40.....	11	600

Freeze-thaw durability studies were made on bars of sulfapave prepared by kneading. The 20-pct-modified sulfapave withstood 300 freeze-thaw cycles and retained 90 pct of its relative dynamic modulus of elasticity. A control sample of asphaltic concrete retained only 35 pct of its relative modulus of elasticity after 300 freeze-thaw cycles.

FLEXURAL FATIGUE

The fatigue behavior of four sulfapave materials was evaluated by flexural beam fatigue testing. The beams were prepared in a manner similar to that used in freeze-thaw and compressive strength measurements. Third-point upward loading

⁶Reference to specific products does not imply endorsement by the Bureau of Mines.

TABLE 4. - Properties of sulfapave materials

Modifier, pct	Composition, pct		Marshall values		Voids, pct	Sp. gr.
	Aggr.	Cement	Stability, lb	Flow, 0.01 in		
10.....	89	11	39,460	5	7.3	2.39
15.....	89	11	23,380	6	7.5	2.36
20.....	89	11	15,540	11	6.5	2.38
25.....	89	11	4,660	19	9.5	2.31
30.....	91	9	3,220	12	3.3	2.39
40.....	89	11	3,110	16	5.4	2.33

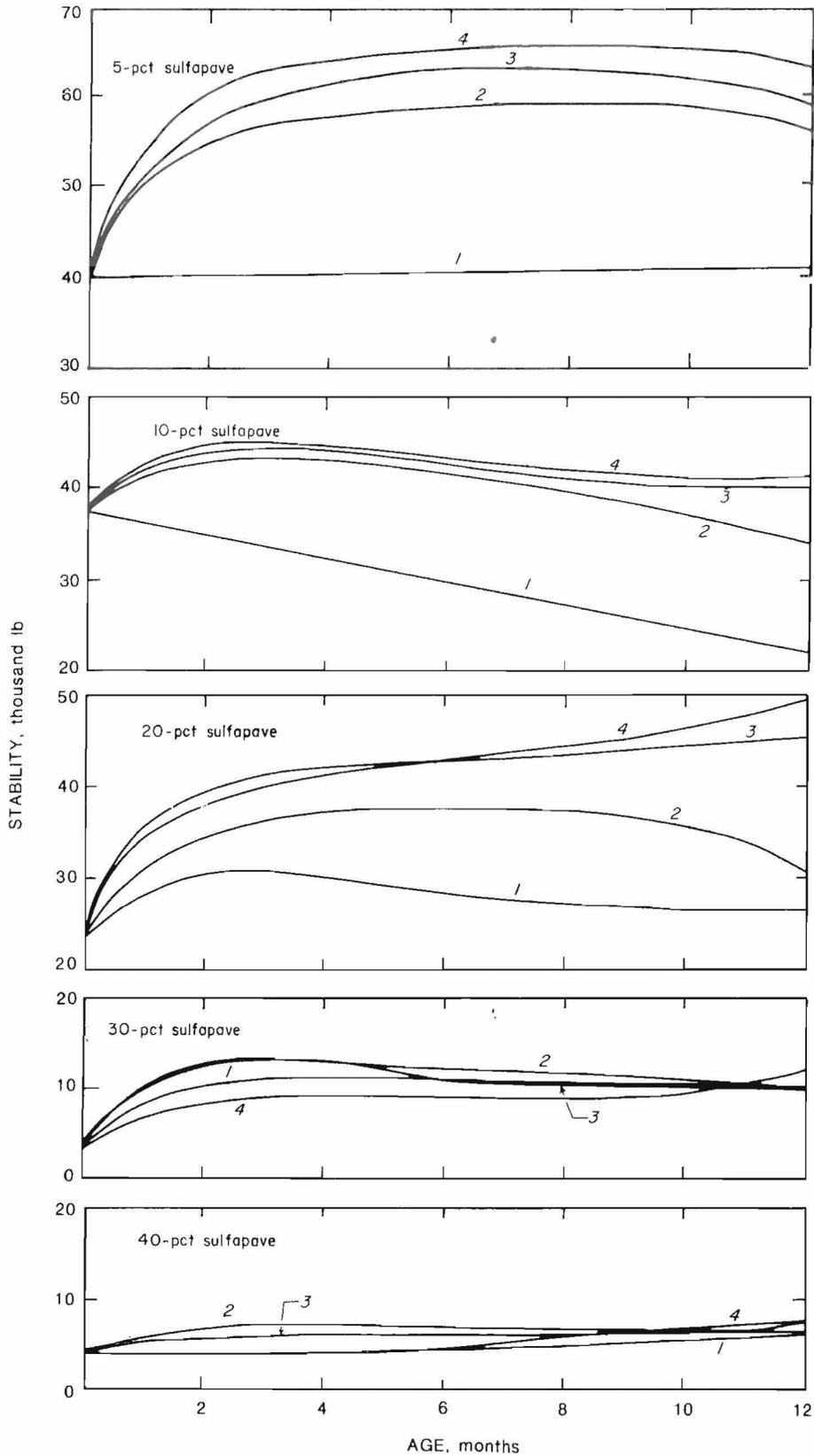


FIGURE 8. - Marshall stability values versus age in corrosion testing of sulfapave. Curve 1, water; curve 2, 10-pct H₂SO₄; curve 3, 20-pct H₂SO₄; curve 4, 60-pct H₂SO₄.

stress-controlled tests were made in accordance with the VESYS IIM Users Manual (41). Since strain increased continually throughout the test, the recorded strain was the stress amplitude divided by the resilient modulus. The beams were tested in a haversine loading mode at a 100-c/min frequency.

The fatigue response for each of the materials is compared in figure 9. Fatigue curves for 5-, 10-, 20-, and 30-pct sulfapave are plotted along with a standard asphaltic concrete mixture. The 5-pct sulfapave resembles asphalt concrete in terms of its fatigue slope. Lee and Klaiber (33) studies on the fatigue properties of conventional sulfur concrete prepared with 5-pct-modified cement showed that the material possessed much higher fatigue life than conventional portland cement concrete (PCC). They found that the sulfur concrete withstood repeated loadings at a much higher percent of modulus of rupture than PCC, and there appeared to be an endurance limit at about 85 to 90 pct modulus of rupture for sulfur concrete compared to 50 to 55 pct for PCC. With increasing modification, 10- to 20-pct sulfapave, the fatigue values obtained were closer to those of the standard AASHTO Asphalt

Concrete. The 30-pct-sulfapave material exhibited superior fatigue properties with essentially a flat S-N relationship.

FIELD TESTING

Small-scale field testing of sulfapave formulations was started in May 1981. A 450-lb batch of 30-pct sulfapave was made by mixing 91 pct dense-graded, 3/8-in quartz aggregate with 9 pct of 30-pct-modified sulfur cement at 135° C in a heated mortar mixer. The test was performed to determine mix and placement characteristics of larger batches of sulfapave materials and to provide sufficient material for monitoring sulfapave durability on exposure to traffic and weathering. The material was placed in a 2-in-deep, 1-ft-wide section across 20 ft of the laboratory roadway. After placement in the roadway, the hot-mix material was compacted with a hand tamper. Finish compaction was done using a small vibratory compactor to compact and level the sulfapave. Mixing, placing, and compacting techniques were similar to those used for hot-mix asphalt paving. No difficulties were found in adapting them to the sulfapave material. Immediately after compaction, the roadway was opened to heavy-duty traffic, which

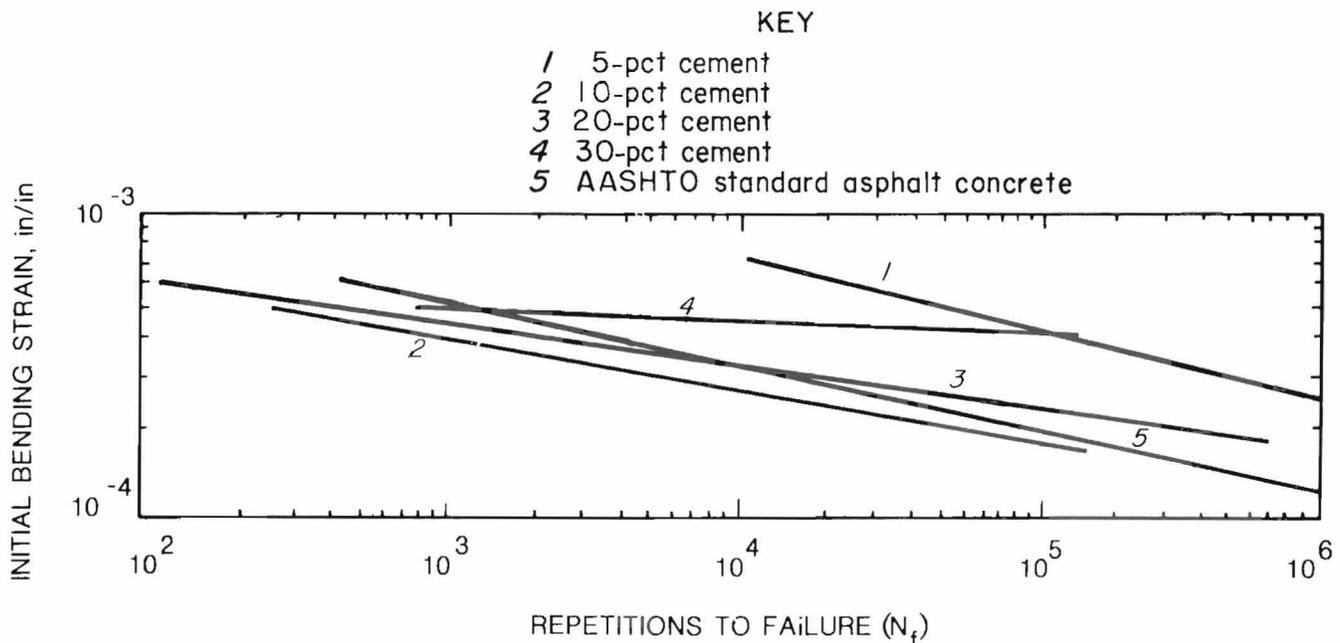


FIGURE 9. - Initial bending strain versus load repetitions for sulfapave.

included truck, forklift, and crane travel. After 2-1/2 years of exposure to traffic and weathering, no signs of deterioration or wear of the pavement have been found.

Two other field tests began in July 1981 using 10-, 20-, 30-, and 40-pct sulfapave formulations prepared in a manner similar to the first test section. Road sections, 2 ft by 2 ft by 2-1/2 in deep, were paved in the laboratory roadway with each of the materials. After 2 years of service, no evidence of rutting or deterioration has been noted.

In the second test, four 2 ft by 2 ft by 3-1/2-in deep slabs were cast and

compacted in a wooden form with each of the materials. The resulting slabs, shown in figure 10, were inserted in a floor section of the TIMET titanium plant in Henderson, NV; the section of concrete floor was being destroyed by chemical corrosion. After 2 yr in service, there has been no evidence of acid corrosion of the slabs or deterioration of the materials from traffic loads.

Although these initial tests indicate the stability of the materials to acidic corrosion and traffic loads, additional larger scale testing of the materials is necessary to establish optimum formulations and durability of the materials.

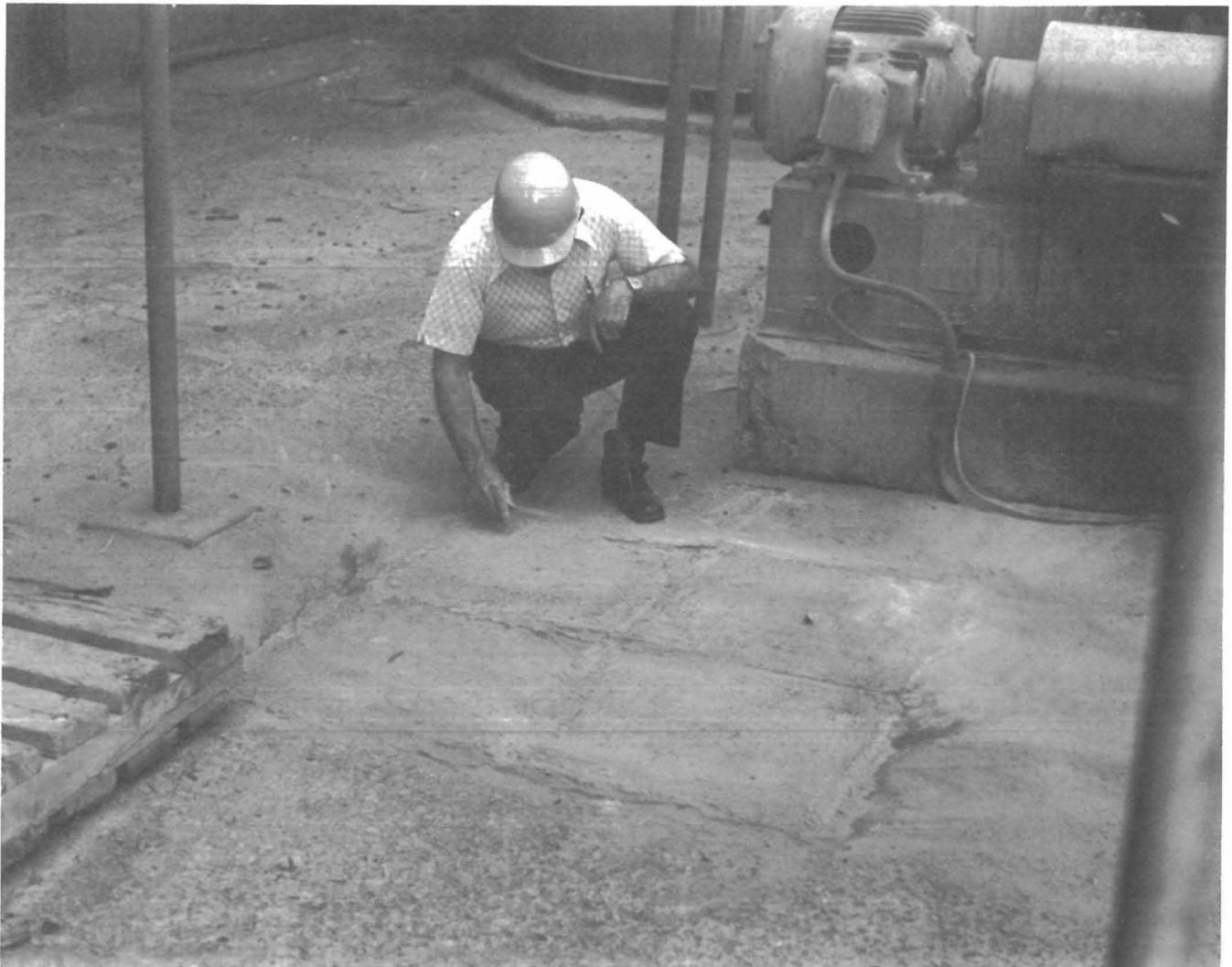


FIGURE 10. - Test sections of sulfapave materials.

SUMMARY AND CONCLUSIONS

Sulfapave materials have been developed with varying physical and mechanical properties by use of mixtures of dense-graded aggregates and sulfur cements prepared by reacting sulfur with 5 to 40 pct chemical modifiers. The chemical and physical properties of various levels of modified sulfur cements were determined. The optimum proportions of the modifiers (DCPD and oligomer) used to prepare the modified sulfur cements were established. Using dense-graded aggregates, the 15- to 30-pct-modified sulfur cements were found most suitable for preparing sulfapave materials with properties similar to asphaltic concrete.

Sulfapave materials have the advantage of being able to be tailored by proper choice of modified sulfur cement and aggregates into materials with properties ranging from rigid PCC to flexible asphaltic concretes. The choice of cement depends on the type of product desired. When high stability, less flexible concretes such as base materials are needed, they can be made using the 5- to

15-pct-modified sulfur cement. If more flexible paving materials are desired, the 25- to 30-pct-modified sulfur cement can be used.

The sulfapave products are suitable for use in most acid and salt corrosive environments. They can be prepared in small lots in conventional construction equipment such as a heated mortar mixer, or in larger amounts in hot-mix asphaltic patch plants. The materials may be laid and compacted into place using conventional paving equipment. The more flexible type sulfapave may be laid without the use of expansion joints. Potential uses for the sulfapave materials include floors, liners for holding and evaporation ponds to contain corrosive and toxic liquors and prevent contamination of the ground waters, and overlay coatings on concrete floors where corrosion is destroying the concrete. Sulfapave materials also have a potential use as a replacement for PCC concrete in bridge decking to eliminate salt corrosion of the concrete and reinforcing steel.

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