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16. Abstract (Limit: 200 words) This program was carried out in three phases and was directed towards (a) establishing a familiarity with basic mix and sample preparation techniques, (b) material characterization, and (c) structural analysis of sulfur concrete mixtures using binders plasticized at 5, 10, 20, 30, and 40 percent. Materials were furnished by the Bureau of Mines Boulder City Engineering Laboratory. Phase I involved the preparation of suitable concrete specimens using the five binders listed above. For mix design operations the Marshall or Hveem procedures for asphaltic mixtures were found to be acceptable. For tests requiring larger size specimens such as unconfined compression and repeated-load creep, the California DOT Test Method 304 is preferred. In Phase II, sulfur concrete mixtures were subjected to a characterization testing program designed to provide input to a linear viscoelastic pavement design system called VESYS IIM. A comparison of pavement performance characteristics such as rutting, roughness, cracking, and serviceability of sulfur concrete materials and asphaltic concretes indicated the former could be expected to behave as well as, or superior to, conventional pavement systems. Phase III investigated the potential of an in-situ characterization device called the Duomorph for monitoring property changes in the five modified-sulfur binders given above. An apparatus and testing procedure is described for measuring moduli of these binders over a range of test temperatures and loading rates.				
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TABLE OF CONTENTS

	Page
List of Tables.....	iv
List of Figures.....	v
1.0 INTRODUCTION.....	1
1.1 Background	1
1.2 Objectives.....	2
1.3 Scope.....	2
2.0 TECHNICAL PROGRAM.....	3
2.1 Materials.....	3
2.2 Phase I - Preliminary Laboratory Effort.....	4
2.2.1 - Sample Preparation and Testing.....	4
2.3 Phase II - Materials Characterization and Analysis of Sulfur Concrete Mixtures.	8
2.3.1 Material Characterization.....	8
2.3.2 Characterization Test Program.....	13
2.3.3 Structural Analysis.....	13
2.3.4 The VESYS System.....	16
2.4 Phase III - The Duomorph Characterization Device.....	20
3.0 DISCUSSION OF RESULTS.....	20
3.1 Phase I - Preliminary Laboratory Effort.....	20

TABLE OF CONTENTS (Continued)

	Page
3.2 Phase II - Material Characterization and Structural Analysis.....	24
3.2.1 Materials Characterization.....	24
3.2.2 Structural Analysis.....	36
3.3 Phase III - Duomorph Testing and Results.....	46
3.3.1 Sample Preparation Problems.....	46
3.3.2 Test Results.....	48
4.0 CONCLUSIONS.....	54
5.0 RECOMMENDATIONS FOR FUTURE WORK.....	55
6.0 REFERENCES.....	56
Appendix A.....	58

LIST OF TABLES

<u>Table</u>		<u>Page</u>
1	Sulfur Concrete Binder and Mix Specifications.....	4
2	Characterization Tests and Properties for Input to VESYS IIM	9
3	Assumed Climatic Temperature Variation for VESYS IIM Input.....	17
4	Summary of Preliminary Engineering Property Test Results of Sulfur Concrete Mixtures.....	22
5	Results of Specific Gravity and Compression Tests on Sulfur Concrete Specimens.....	25
6	Results of Freeze-Thaw Tests in Sulfur Concrete Mixtures.....	27
7	Summary of Stiffness Moduli and Beta Values.....	35
8	Summary of Permanent Deformation Parameters, Alpha and G _{Nu}	37
9	Resilient Modulus Versus Temperature Degree of Plasticization on Unfilled Binder.....	46

LIST OF FIGURES

<u>Figure</u>		<u>Page</u>
1	Gradation No's. 1, 2, 3 and 4 Used in Phase I Sulfur Concrete Mix Designs.....	6
2	Gradation No. 5 Used in Phase I Sulfur.....	7
3	Stress-Strain of Incremental Static Test Series...	10
4	Test Program Using Bureau Furnished Sulfur Concrete Samples.....	14
5	VESYS Analysis Schematic.....	15
6	Compressive Strength vs. Percent Binder (35/65)...	23
7	Initial Bending Strain vs. Load Repetitions to Failure for Sulphur Concrete Mixes.....	28
8	Comparative S-N Curves for Sulfur Concrete and Portland Cement Concrete.....	32
9	Creep Curves for Sulphur Concrete Mixtures and Laboratory Standard Asphaltic Concrete.....	34
10	Permanent Deformation versus load Duration for Sulfur Concrete Mixtures and Laboratory Standard Asphaltic Concrete.....	38
11	Present Serviceability Loss with Time for Thin Pavement Surfaces (2 Inches) for Sulfur-Concrete and Laboratory Standards Asphaltic Concrete.....	39
12	Present Serviceability Loss with Time for Thick Surfaces (6 Inches) for Sulfur Concrete and Laboratory Standard Asphaltic Concrete.....	40
13	Change in Slope Variance (Roughness) with Time (Age) for Sulfur Concrete and Laboratory Standard Asphaltic Concrete.....	42

LIST OF FIGURES (Continued)

<u>Figure</u>		<u>Page</u>
14	Increase in Cracking Damage Index with Times for Thin Pavements of Sulfur Concrete and Laboratory Standard Asphaltic Concrete.....	43
15	Increase in Cracking Damage Index with Time for Thick Pavements for Sulfur Concrete and Laboratory Standard Asphaltic Concrete.....	44
16	Increase in Rut Depth with Age of Sulfur Concrete and Laboratory Standard Asphaltic Concrete.....	45
17	Test Sample in Fiberglass Cavity Containing Duomorph.....	49
18	Internal View of Duomorph Test Fixture and Components.....	49
19	Duomorph Test Fixture.....	50
20	Duomorph Test Set Up with N ₂ Bottle and Readout System.....	50
21	Comparison of Modulus Versus Temperature From Duomorph and Resilient Modulus Tets at Various Plasticized Sulfur P/S Ratios.....	51
22	Effects of Duomorph Excitation Frequency on Modulus Versus Temperature and P/S Ratio of Plasticized Sulfur Binders.....	52

1.0 INTRODUCTION

1.1 Background

The Bureau of Mines has been active in the development of modified sulphur concrete systems as part of its program to find new uses for a potential excess of sulfur that is anticipated from pollution abatement sources [1]. Previous work involved the use of sulfur in conjunction with asphalt for: (a) use with sands and other marginal aggregates in sand-asphalt-sulphur (SAS) systems [2], (b) use as an asphalt extender in sulfur-extended-asphalt (SEA) mixtures [3] and (c) use as an additive for the recycling of old bituminous pavement systems [4]. TTI has supported the Bureau's Boulder City Engineering Laboratory (BCEL) sulfur utilization activities, primarily, in the areas of freeze-thaw and flexural fatigue testing.

More recently the Bureau has been involved in the development of specialized sulfur concretes (similar to portland cement concrete) for use in applications involving highly corrosive acid and salt environments [5]. The chemical, physical and mechanical characteristics of chemically-modified sulfur cement and concrete are described in a paper by McBee, et al., [6].

Whereas the Bureau's research has included the measurement of basic strength, durability and corrosion resistance of paving type materials, little has been done to establish in-service performance predictions. To carry out such an evaluation, characterization tests are required which are compatible with currently available pavement design computer codes. The research described herein was aimed at generating a linear viscoelastic pavement analysis for the prediction of in-service performance such as rutting potential, fatigue crack susceptibility, slope variance (rideability) and serviceability. The analysis discussed in this report incorporates the structural and behavioral characteristics of several "lean," modified-sulfur concretes which were prepared by mechanical compaction to meet specifications for asphaltic type paving materials.

In addition a device, still in the developmental stages, called the Duomorph was investigated for possible use as an

"age-meter" or an in-situ mechanical behavior characterization instrument. The potential for using this device to detect allotropic changes in the sulfur phase of the cement during cool-down from the liquid melt was also studied.

1.2 Objectives

The primary objective of this research was to assess the structural performance of plasticized sulfur concrete mixtures using advanced behavioral characterization tests compatible with state of the art pavement design computer codes. A second objective was to investigate the use of the Duomorph as a means of monitoring changes in material properties with loading rate and temperature.

1.3 Scope

This report presents the results of the laboratory and analytical activities generated in the accomplishment of the objectives stated above. The laboratory effort involved a series of characterization tests on five different Bureau of Mines plasticized sulfur concrete mix designs. Specific tests include:

- (1) Compression
- (2) Freeze-thaw
- (3) Fatigue
- (4) Repeated Load Creep

The analytical phase of the study employed these test results for the prediction of in-service pavement performance in terms of:

- (1) Rutting Potential
- (2) Slope Variance (surface smoothness)
- (3) Fatigue Crack Resistance
- (4) Serviceability

Finally, the potential of the Duomorph was investigated to monitor stiffness changes in plasticized sulfur binders with loading rate and temperature. The ability to detect allotropic forms of sulfur binders during cool-down from the melt was also studied.

2.0 TECHNICAL PROGRAM

The overall program was conducted in three phases. Phase I utilized binder materials furnished by the Bureau's Boulder City Engineering Laboratory (BCEL) to familiarize TTI personnel in the preparation of sulfur concrete mixtures. Mixtures were prepared with modified-sulfur binders using varying plasticizer contents. Both rigid (5 percent plasticizer) and flexible binders were used in an attempt to establish sample preparation procedures especially for the larger specimens to be used in the characterization testing phase of the program. Several different aggregate gradations were also incorporated into the mixtures prepared in this phase of the program.

In Phase II, sulfur concrete samples were subjected to more in-depth characterization tests for incorporation into a theoretical analysis of the materials' in-service performance. These concrete specimens were prepared by BCEL with binders having the same plasticizer to sulfur ratios as used in Phase I. A state-of-the-art linear viscoelastic computer code was employed in this analysis.

Phase III used the binders from Phase I to exploit the capability of the Duomorph, a transducer in the development stages at TTI, to monitor binder property changes over a range of temperatures and loading rates.

2.1 Materials

At the outset of the program five plasticized sulfur binder materials with 5, 10, 20, 30 and 40 percent plasticizer, respectively, were prepared by the Bureau using their mixed-modifier process. The materials were formulated using a mixture of Dicyclopentadiene and oligomers of Cyclopentadiene as described in Reference 5. The materials were shipped to TTI in 10-one gallon cans (i.e., 2 gallons of each binder) and utilized in Phases I and III.

In addition, sulfur concrete samples were prepared by BCEL using the same five binder formulations. These were prepared using the Marshall Design Method (ASTM D1559-75) for asphaltic pavements. A quartz aggregate sized to meet Type IVb Asphalt Institute specifications was incorporated into these mixtures.

These samples were furnished to TTI in five separate shipments for use in Phase II. Each shipment contained eight (8) concrete bars - 3 in. (7.6 cm) x 3 in. (7.6 cm) x 15 in. (38.1 cm). The binder identifications and concrete mix specifications are given in Table 1.

Table 1. Sulfur Concrete Binder and Mix Specifications

<u>Sample Number</u>	<u>Binder Content %</u>	<u>Plasticizer Content (%)</u>	<u>Binder O/DCPC Ratio*</u>
M92	11	5	50/50
M93	11	10	60/40
M94	9	20	75/25
M95	9	30	75/25
M96	9	40	75/25

*Oligomer to Dicyclopentadiene ratio

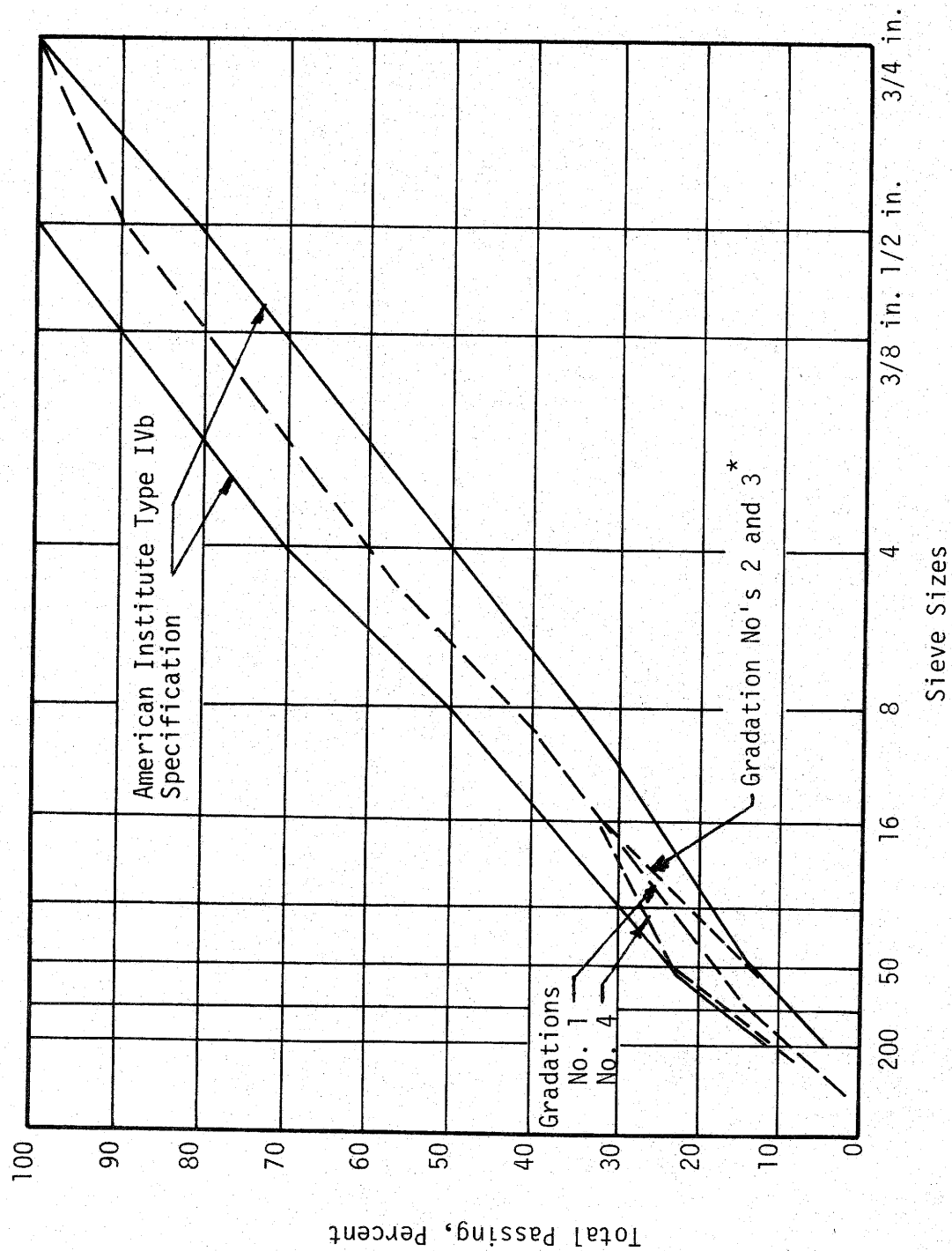
2.2 Phase I - Preliminary Laboratory Effort

2.2.1 Sample Preparation and Testing. Using the five modified-sulfur binders furnished by the Bureau, preliminary mix preparation activity was undertaken to provide TTI personnel with experience in handling, sample preparation and testing of sulfur concretes.

Initially 6 in. (15.2 cm) diameter x 12 in. (30.4 cm) high samples were prepared for compression testing. To conserve material test specimens 4 in. (10.2 cm) in diameter and 8 in. (20.4 cm) high had to be used. The major requirement in this activity was to maintain temperature control of all hardware during mold filling and compaction. The latter was accomplished using the California DOT Test Number 304 (1978) for bituminous mixtures. The first samples were highly porous, and non-homogeneous, especially on the surfaces in contact with the mold walls. This required that the molds be filled, tamped and compacted in not greater than 3 in. (7.6 cm) lifts. After several trials, sample preparation problems were resolved except for a slight depression at the free end created by cure shrinkage. This was compensated for by making the samples 1 in. (2.5 cm) longer, cutting off and capping the ends. This activity utilized most of the Bureau-furnished binder material on hand. It was therefore decided that BCEL would furnish the samples for the characterization effort in Phase II.

A number of different mix designs, using two rigid binder systems furnished by Chemical Enterprises of Odessa, Texas were tested in which the aggregate size fractions were varied to adjust the air voids in the final compacted sample to range between 3-5 percent as recommended by the Bureau. Both of these binders had a 5 percent plasticizer content but differed with respect to their oligomer to DCPC (O/DCPD) ratios which were 35/65 and 50/50, respectively. Preliminary tests were run to determine if the California DOT Test No. 304 procedure could be used to prepare concrete specimens with rigid as well highly plasticized binders.

Basic properties (1) Compressive Strength, (2) Air Voids Content and (3) Rice Specific Gravity versus binder content and five different gradations were measured. Each gradation system was evaluated at 3 different binder contents. Gradation No.'s 1, 2, 3 and 4 utilized an Asphalt Institute IVb specification (See Figure 1), with variations in the amounts of fines (minus No. 16 sieve) material. Gradation No. 5 was prepared by changing the aggregate fractions to represent a more open-graded system typical of an Asphalt Institute Type IIb (See Figure 2). In gradation No. 3, fly ash (18 percent) was used in place of the minus No. 50 sieve, crushed limestone. Gradation No. 4 was used in mixes which employed the binder with an O/DCPD ratio of 50/50. All of the others used the binder with a 35/65 ratio.



* Gradation No. 3 used 18 percent fly ash for minus 50 fines.

Figure 1. Gradation No's. 1, 2, 3 and 4 Used in Phase I Sulfur Concrete Mix Designs.

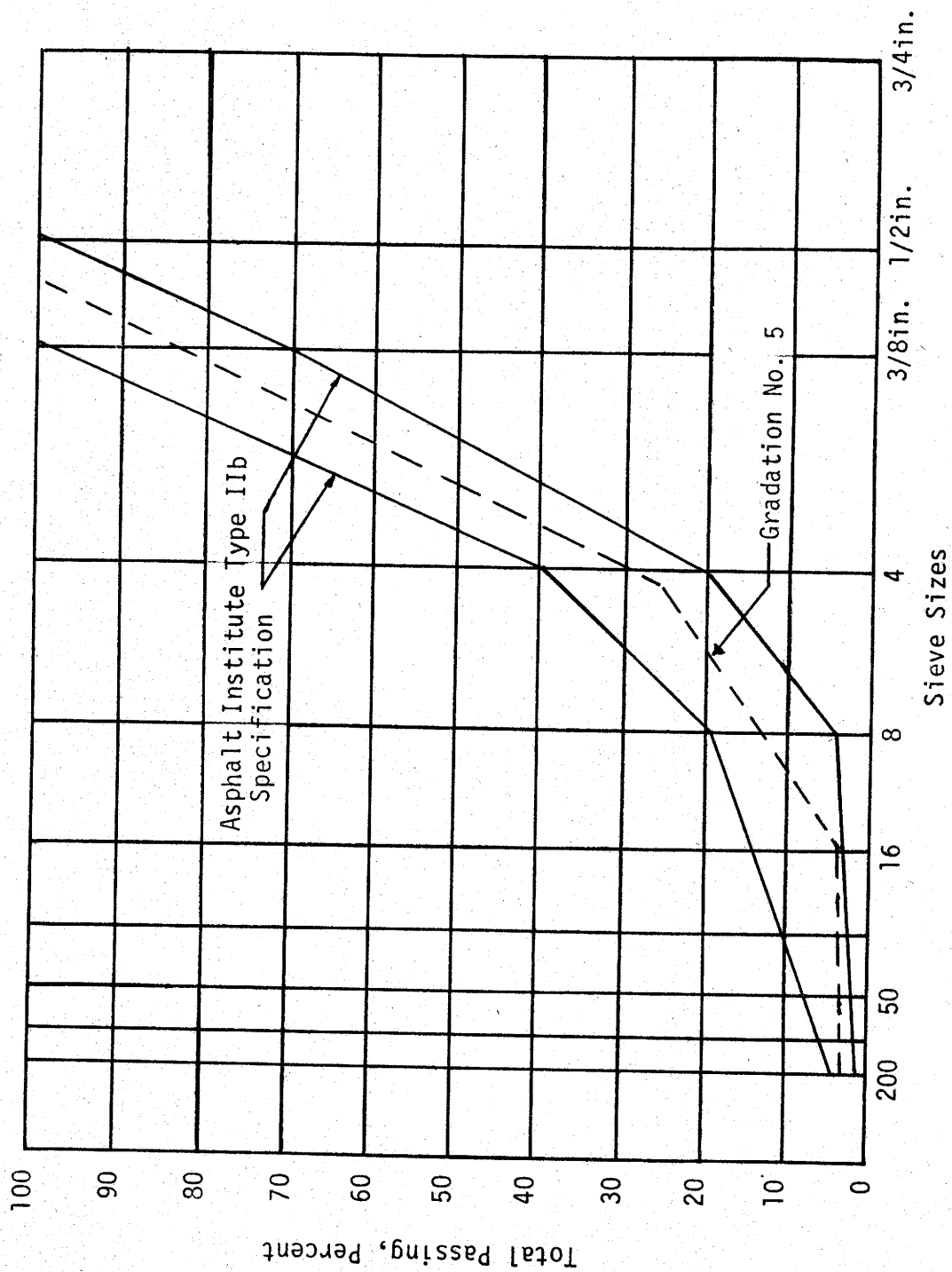


Figure 2. Gradation No. 5 Used in Phase I Sulfur Concrete Mix Designs.

2.3 Phase II - Materials Characterization and Analysis of Sulfur Concrete Mixtures

2.3.1 Material Characterization. Two categories of mechanical properties are required for structural analysis of paving materials: primary response properties and distress properties. The primary response properties define the response of the materials to given loads and environments. These properties are in the form of elastic or viscoelastic characteristics which may exhibit non-linear behavior because of previous load histories or due to plastic effects and stress/strain dependencies of the response coefficients. The distress properties are those defining the capability of the material to withstand an imposed load. These are usually determined from special laboratory tests simulating the type of stresses and range of climatic conditions anticipated during service. The analysis utilized in this program was a linear viscoelastic computer code designated VESYS IIM [7,8], an acronym for Viscoelastic Systems.

The material properties used in the VESYS programs are given in Table 2 along with the test conditions necessary for their determination. These tests involve the generation of the following response characteristics.

- (1) Creep Compliance
- (2) Permanent Deformation μ_j and a_j
- (3) Fatigue Constants K_1 and K_2

The parameters are obtained using two different types of loading patterns; dynamic (repeated) and incremental-static-dynamic. The latter loading pattern is shown in Figure 3. The materials properties listed above were determined at 33, 68, and 104°F (1, 20 and 40°C). Short descriptions of the three response characteristics are given below.

1. Creep Compliance: The primary response properties require determination of the creep compliance function for rate-dependent materials and elastic compliance for rate-independent materials. The rate-dependent properties are obtained by conducting creep tests. Input to the VESYS program for primary response only requires use of the 1,000-second creep test from the "incremental static test series" (Figure 3). Values of the creep compliance, as described by Equation 1 are plotted versus time on log-log paper:

Table 2. Test Types for Determining Materials Properties
For Input to VESYS IIM

TEST DESCRIPTION		MATERIAL PROPERTIES DETERMINED FOR EACH LAYER i		
BOUNDARY CONDITIONS	TYPE LOADING	CREEP COMPLIANCE $D(t)_i, i = 1,2,3$	PERMANENT DEFORMATION $\mu_i, \epsilon_i, i = 1,2,3$	FATIGUE $K_1, K_2, i=1$
DIRECT COMPRESSION CYLINDER	DYNAMIC		X	
	INCREMENTAL STATIC- DYNAMIC	X	X	
* INDIRECT TENSION	DYNAMIC		X	X
CYLINDER, CORE	INCREMENTAL STATIC- DYNAMIC	X	X	X
FLEXURE BEAM	DYNAMIC ONLY			X

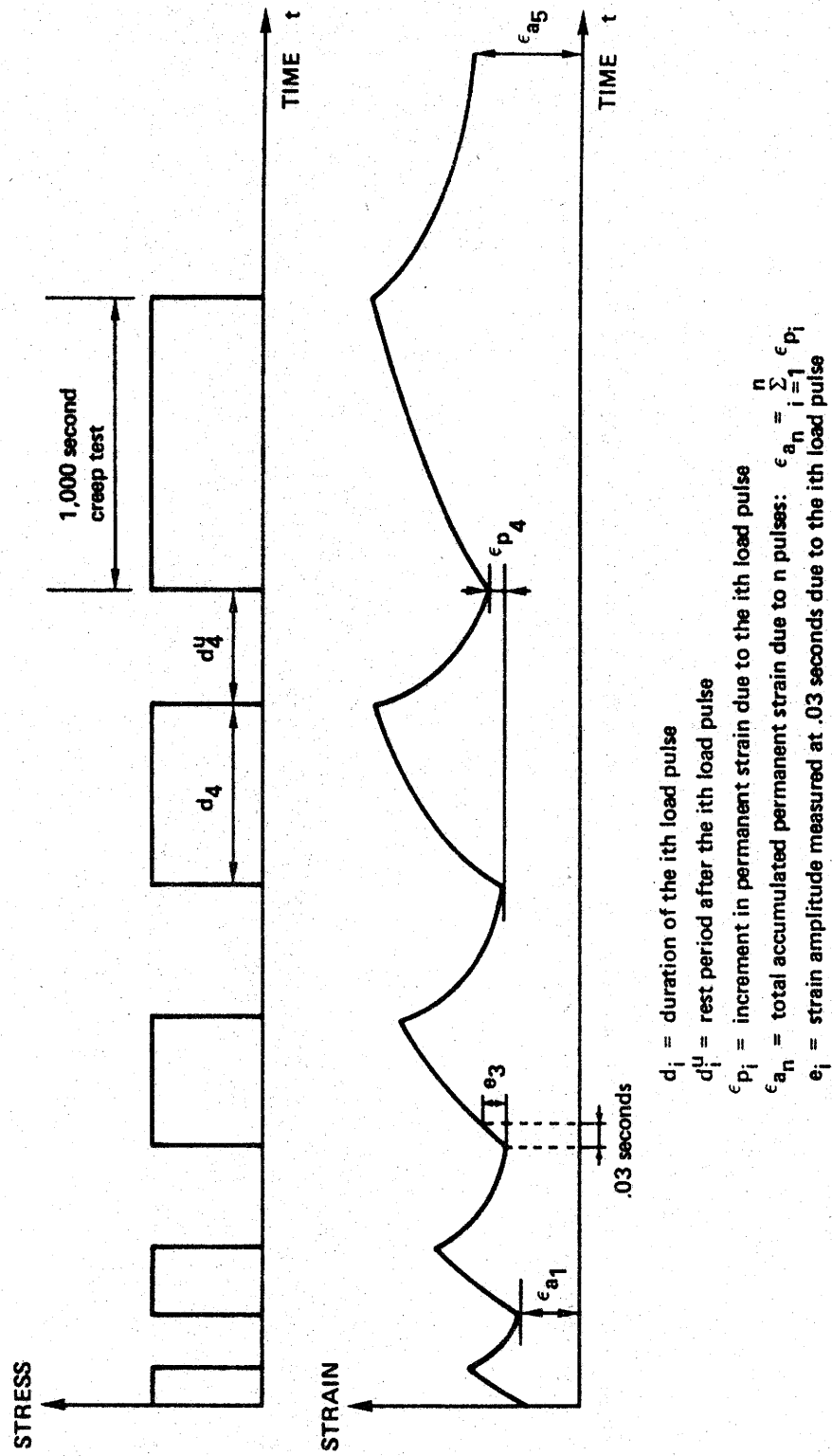


Figure 3. Stress-Strain of Incremental Static Test Series.

$$D(t) = \frac{\epsilon_{zz}(t)}{\sigma_{zz} - 2\mu(t)\sigma_{rr}} \quad (1)$$

where: $D(t)$ = modular creep compliance function

σ_{zz} = axial load in a tension or compression test
with or without confinement pressure,

σ_{rr} = confinement pressure,

ϵ_{zz} = axial strain

$\mu(t)$ = Poisson's ratio as a function of time.

Care should be taken to assure that test results reflect the effects of stress state, temperature, moisture content and conditions corresponding to those anticipated by the pavement. A sufficient number of tests at different temperatures are run to establish the master creep compliance curves and, hence, the time-temperature shift parameters to permit the extension of the results to the prediction of behavior over a wide range of climatic conditions.

2. Permanent Deformation: The permanent deformation properties as indicated in Table 2 are μ and α for each layers $i = 1, 2, 3$. These properties were obtained by conducting the Incremental Static-Dynamic Test on 3 in. (7.6 cm) x 3 in. (7.6 cm) x 6 in. (15.2 cm) long specimens. Since these properties are dependent on in-situ stress conditions and local environments, they should be determined on specimens subjected to realistic in-situ stress states and at the average moisture content and temperatures expected in the field. The properties may be computed from the results of the dynamic test series for

all materials or from the results of the simplified incremental static test series for those materials which exhibit predominant amounts of viscous flow. The permanent deformation properties, μ and α are defined in the following relationships:

$$\mu = \frac{I_s}{\epsilon_r} \quad \text{and} \quad (2)$$

$$\alpha = 1-s \quad (3)$$

where: I = intercept after one of the log-log plot of permanent strain versus load repetitions

s = slope of the log-log plot of accumulated strain versus load repetitions

ϵ_r = the resilient strain.

(3) Fatigue: Fatigue properties are represented by the parameters K_1 and K_2 described by Equation 4. These properties are obtained by conducting repeated load flexural tests on beam specimens at various temperatures

$$N_q = K_{1q} (1/R_q)^{K_{2q}} \quad (4)$$

where: N_q = Number of loads to failure under temperature and strain conditions of the q th time interval

R_q = General radial strain response

K_{1q} and K_{2q} = Material fatigue properties.

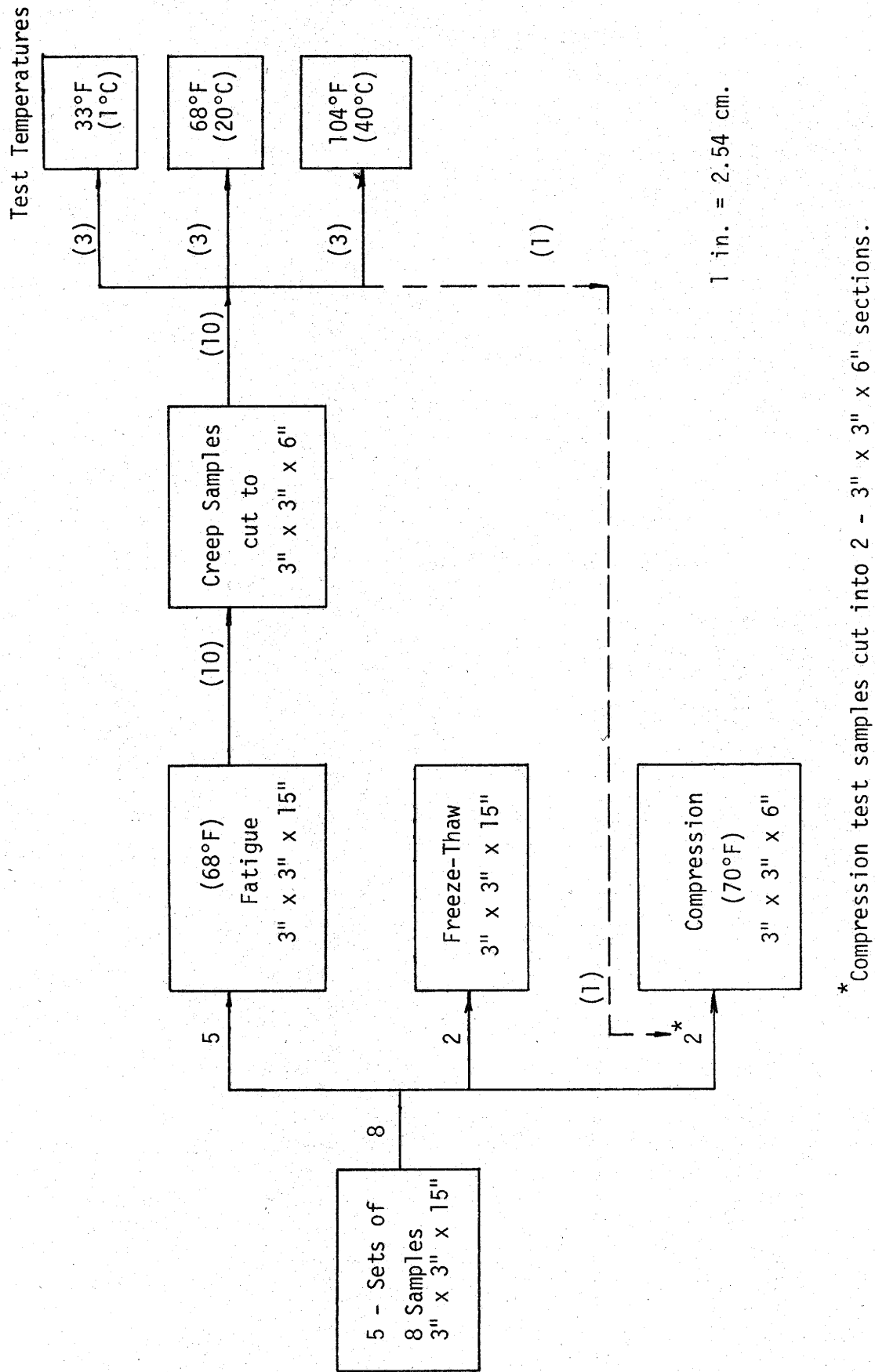
Fatigue relationships in the form of Equation 4 were determined for the five sulfur concretes tested. The tests were performed at 68°F (20°C). The VESYS program provides a mechanism to shift the material properties K1 and K2 for changes in temperature. Since the effects of temperature on K1 and K2 were not determined in this study, a standard shift procedure was used to account for climatic and monthly temperature variation. These shift factors were developed for fatigue data based on the behavior of asphalt concrete mixtures.

2.3.2 Characterization Test Program. Eight sulfur concrete samples 3 in. (7.6 cm) x 3 in. (7.6 cm) x 15 in. (38.1 cm), representing each of the five binders shown in Table 1 were shipped to TTI by the Bureau of Mines for testing and evaluation in accordance with the test program shown in Figure 4.

Seven samples from each set of eight were used; five for constant stress-amplitude fatigue and two for freeze-thaw tests. The remaining sample was cut into two 3 in. (7.6 cm) x 3 in. (7.6 cm) x 6 in. (15.2 cm) sections for compression testing. Upon completion of the fatigue tests, the broken samples were cut into ten 3 in. (7.6 cm) x 3 in. (7.6 cm) x 6 in. (15.2 cm) units for repeated load creep tests. This permitted the creep tests to be run in triplicate at 33°F (1°C), 68°F (20°C) and 104°F (40°C). The remaining sample was used in the compression test to permit tests to be run in triplicate. Stress levels in the fatigue tests were arbitrarily selected to provide the best definition of the respective fatigue curves for each binder.

2.3.3 Structural Analysis. The approach used to evaluate the structural capability of the plasticized sulfur mixes was based on the VESYS IIM structural system [7]. With this system the plasticized sulfur binders can be compared to conventional binders using the above characterization test results and existing data for both flexible and rigid pavements.

Five (5) types of plasticized sulfur-aggregate mixtures as furnished by the Bureau of Mines (See Section 2.1) were analyzed. These plasticized sulfur mixtures were evaluated in the VESYS IIM structural system along with a conventional asphalt concrete containing an aggregate matrix similar to that used in the Bureau-furnished mixtures. Figure 5 shows the schematic of the proposed analysis.



* Compression test samples cut into 2 - 3" x 3" x 6" sections.

Figure 4. Test Program Using Bureau Furnished Sulfur Concrete Samples

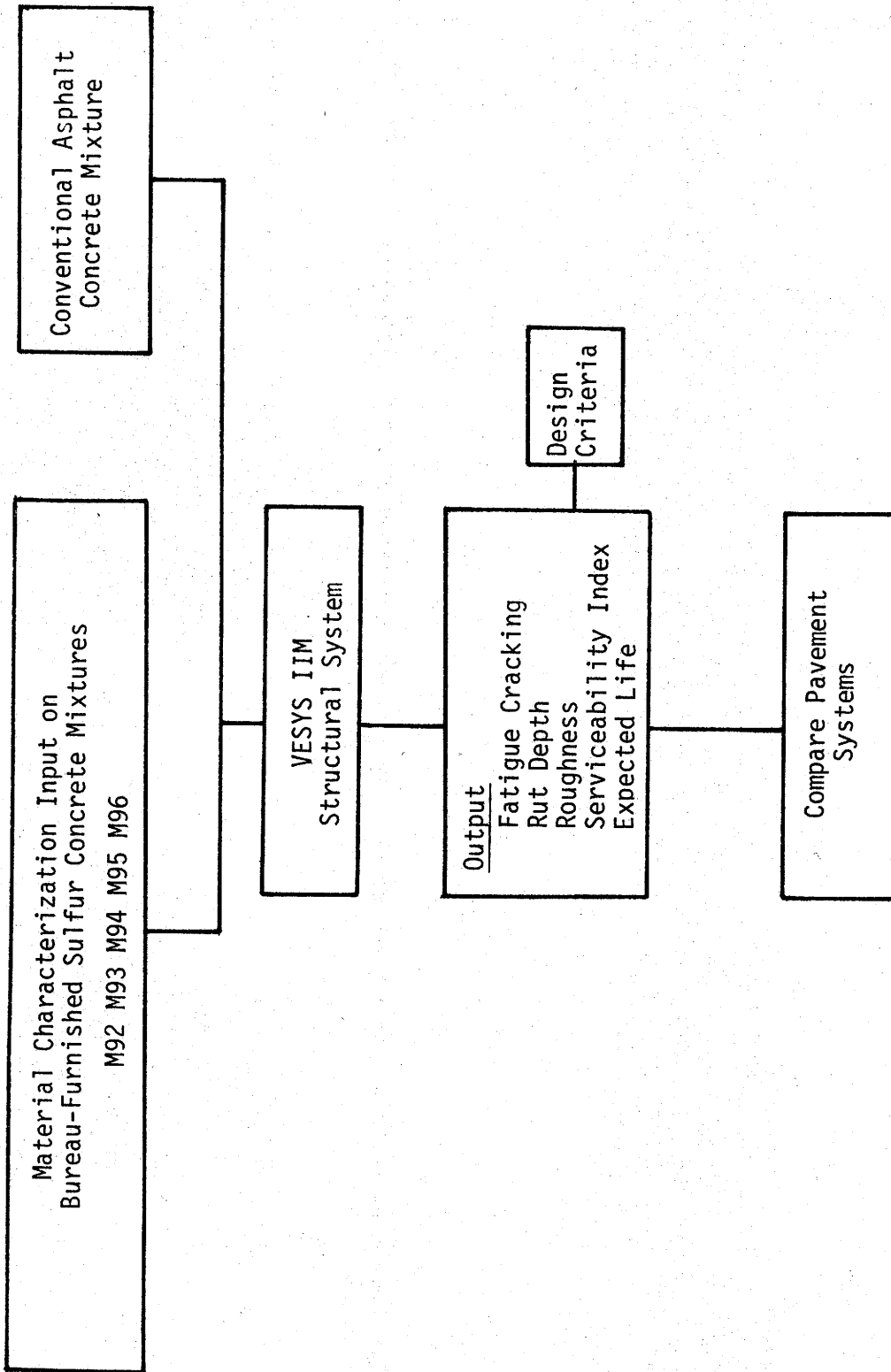


Figure 5. VESYS Analysis Schematic.

The analysis can be run for a wide variety of pavement geometries and climatic regions for each of the five mix designs once their respective material characterization has been completed. The objective was to evaluate the five plasticized sulfur-aggregate mixtures against a conventional asphalt concrete mixture for several different climatic regions. This analysis considered three climates (cold, moderate and warm).

Table 3 is a summary of the mean monthly temperatures used in the three climatic regions. Generally, the cool climatic region is approximately 10°F (5.5°C) cooler than the moderate region which, in turn, is approximately 20°F (11°C) cooler than the warm region. This comparative climatic triad was utilized in this program to evaluate temperature effects on pavement performance.

2.3.4 The VESYS System. The core of the VESYS II program is an n-layer linear viscoelastic program which calculates stress, strains, and displacements and their variances at selected points within the layers. The calculated results are then used together with empirical relations developed either in the laboratory or by field observations to predict various forms of distress and the associated Present Serviceability Index (PSI) and its variance using the AASHTO equation [(8)]:

$$\begin{aligned} \text{PSI} = & 5.03 - 1.91 \log_{10} (1 + \text{SV}) \\ & - 0.01 (\text{C} + \text{P})^{1/2} - 1.38 (\text{RD})^2 \end{aligned} \quad (5)$$

where SV = slope variance
C = cracking
P = patching
RD = rut depth

This relation of distress to performance can be expected to be most valid in the climatic region represented by the AASHTO Road Test and would probably fail to predict performance with equal reliability in some other climatic region. Because of this, the above equation simply represents a criterion of performance which may or may not bear much relation to pavement performance in a given region. The program is nevertheless a

Table 3. Assumed Climatic Temperature Variation for VESYS IIM Input.

Month	Temperature, °F		
	Cool	Moderate	Warm
1	11.0	22.0	41.0
2	14.0	22.5	43.5
3	15.5	25.5	45.5
4	34.0	46.0	65.5
5	51.0	61.0	30.0
6	59.0	69.0	89.5
7	63.5	73.0	93.5
8	65.0	75.0	95.0
9	61.5	72.0	91.0
10	57.5	67.0	87.5
11	34.0	44.0	64.0
12	28.5	39.0	59.5

$$^{\circ}\text{C} = 5/9 (^{\circ}\text{F} - 32)$$

valuable tool for stress and distress analysis which is superior in its capabilities to any other such tool presently available in the pavement structural design and analysis field.

VESYS II allows for input of various classes and loads and monthly temperature variations. It employs time-temperature shift methodology based on thermo-rheologically simple behavior. The resulting shift factors are developed from creep tests over temperature ranges similar to those expected in the field. The complete characterization is expressed in reduced time which provides for the change in material properties of a linearly viscoelastic, "thermorheologically simple" material as its in-service temperature changes.

The rutting model in VESYS expresses rut depth as a function of stress, duration of loading, temperature and moisture. The pavement rutting characteristics are computed in VESYS using layer theory in conjunction with a laboratory determined permanent deformation law for each pavement layer. Equation 6 represents the form of the incremental deformation model.

$$f(N) = \mu N^{-\alpha} \quad (6)$$

where $f(N)$ = the fractional increase in strain with each load repetition, N

μ = from Equation 2

α = from Equation 3

The model accounts for real world variability by treating the elastic and viscoelastic moduli, load amplitude, load duration and load repetitions, N , as random variables described by their respective means and variances. Taking the approximate moment of Equation 6, after integration over N cycles, yields the desired rut depth equation for mean rut depth and variance.

The fatigue model is a phenomenological model which is used to predict the extent of cracking based on Miner's hypothesis which states that fatigue failure is reached when

$$\sum_{i=1}^n \frac{n_i}{N_i} = 1 \quad (7)$$

where n_i = number of load applications at stress level i

and N_i = number of load applications at stress level i which would cause cracking.

The number of applications to cause cracking at a certain tensile strain is in turn given by Equation 4.

The expected cracking damage is expressed as a dimensionless index where the crack as initiated appears on the pavement underside. Using stochastic solutions to Miner's equation and in turn developing cumulative distribution functions, the expected cracked area is determined and is expressed in square feet of cracked area per 1,000 square feet of pavement.

The roughness model is computed as the variation in deformation along the longitudinal profile of the roadway. In this model, accumulated rut depth at any time along the wheel path is assumed to vary as a result of material variability and varying construction practices. A pavement's roughness is expressed by slope variance.

2.4 Phase III - The Duomorph Characterization Device

An attempt was made to use a characterization device, currently under development at the Mechanics and Materials Research Center (MMRC) at Texas A&M, as a means of monitoring property changes in plasticized sulfur binder systems. This device, the Duomorph, has been used to measure viscoelastic properties of asphaltic concretes in both field and laboratory environments [9]. It is being developed for use either as a surface or embedded transducer on materials with moduli ranging from 10 to 10^6 psi. The small deformations under which these measurements are obtained permit subsequent analysis to proceed on the basis of linear viscoelastic behavior.

The transducer consists of two piezo-electric crystals bonded together so that, when excited electrically, they bend about their central plane. The amount of bending is a function of the stiffness of the Duomorph, the excitation voltage and the modulus of the material being tested. By varying the voltage and bending frequency, a characterization of the rate dependent behavior of a material can be accomplished.

A nomograph has been generated to facilitate this data reduction. A copy of Reference [9], discussing some of the design and analysis aspects of the Duomorph along with calibration and data reduction procedures, is appended to this report (See Appendix A). Also treated in the paper are the results of data taken on samples obtained from an asphaltic concrete pavement in Texas.

3.0 DISCUSSION OF RESULTS

3.1 Phase I - Preliminary Laboratory Effort

The sample preparation procedures used for sulfur concretes were dictated by sample size. For mix design testing, samples may be prepared using the methods outlined in the Marshall Design Method (ASTM D-1559-75) or the Hveem Design Method (ASTM D-1560-71). These specimens are 4 in. (10.2 cm) in diameter and 2 1/2 to 3 in. (6.4 to 7.6 cm) in height and can also be used for resilient modulus and indirect tension tests. When larger samples are prepared, that is, with thicknesses greater than 3

in (7.6 cm) the molding must be carried out in multiple lifts. In this case some degree of tamping is necessary to minimize any localized weak zones in the vicinity of the interface between two lifts. The method found to be most suitable for these larger specimens was the California DOT Test No. 304 - "Method of Preparation of Bituminous Mixtures for Testing."

This method introduces the mix to the mold in no greater than 3 in (7.6 cm) lifts with the application of 20 roddings per lift in the center and around the rim of the mold. This is followed by 20 tamping blows of 250 psi pressure using a kneading type compactor. After the mold is filled and each lift has been suitably rodded and tamped the samples are placed in a 250°F (121°C) oven for at least 2 hours prior to applying a 12,600 lb. leveling load. This method was used to prepare samples 4 in. (10.2 cm) in diameter and 8 in. (20.4 cm) high for compression tests. It was found that the California method worked equally well with rigid binders (5 percent plasticized) except the 12,600 lb. leveling load may be omitted.

Almost all of the binder material furnished by BCEL was used in the preliminary sample preparation activity discussed above. About 500 cc of each of the five binders systems was set aside for use in the Duomorph studies in Phase III. The remainder of the work in Phase I was accomplished using two rigid binders; 5 percent plasticizer with O/DCPD ratios of 35/65 and 50/50, respectively. These were incorporated into the five mix designs discussed in Section 2.1 to determine the relative effects of different aggregate gradations and binder contents and the appropriateness of the California DOT Test No. 304 method for preparing these samples. The results of these tests are shown in Table 4 and Figure 6.

Air voids in the resulting mixes ranged from 0.5 to 4.5 percent. Compression strengths were consistent with that reported by BCEL for dense graded rigid concrete systems [6]. The lowest compressions strengths were exhibited in the open-graded mixture using Gradation No. 5 (Type IIb). This was due to the porosity inherent in open-graded systems which tends to permit the hot binder to seep through the samples during molding and compaction to produce a non-homogenous composite. The primary reason for using an open-graded design was to increase the air

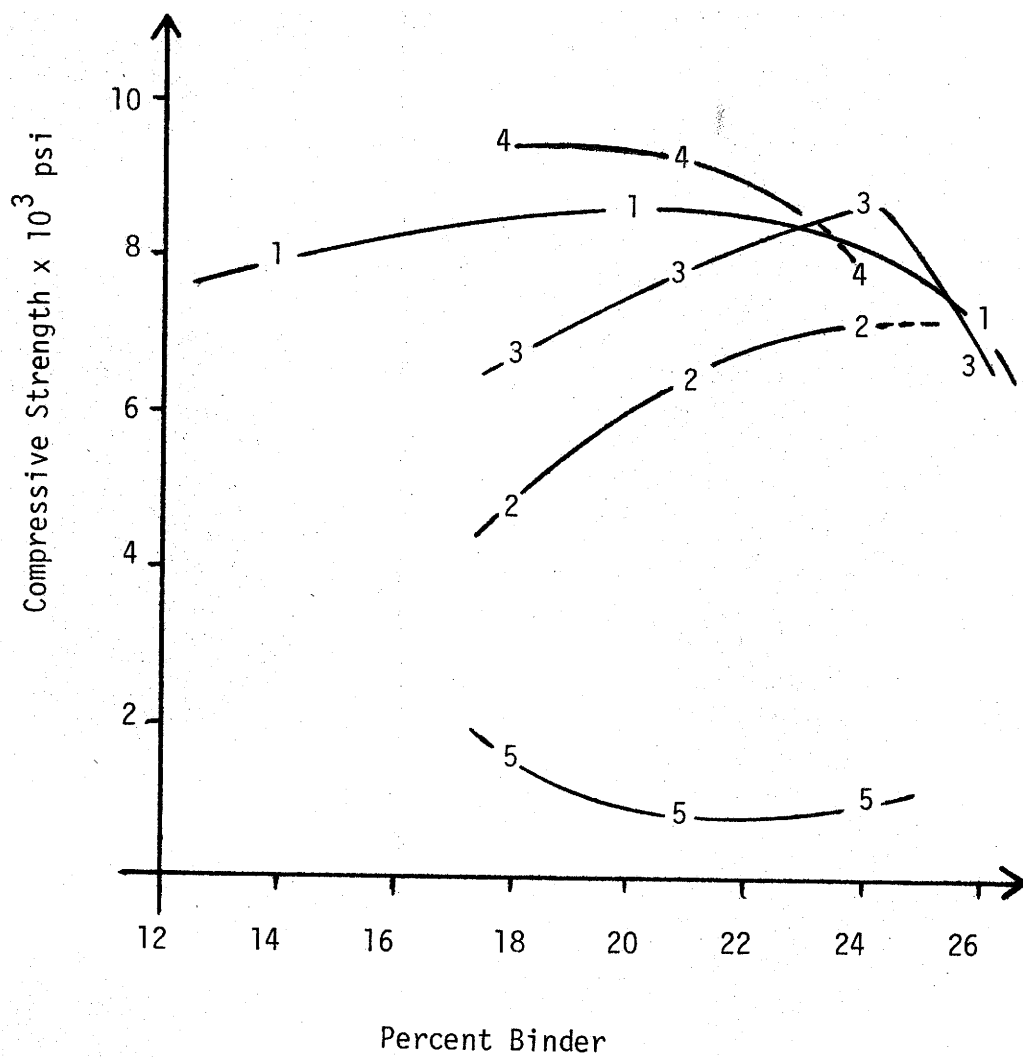


Figure 6. Compressive Strength vs. Percent Binder
(O/DPCD = 35/65).

Note: Numbers in the plot indicate gradation
used in the mixes (see Figures 1 and 2).

voids which, except for mixes using Gradation No. 1, were either outside or barely within the 3 to 5 percent range recommended by the Bureau. The poor compressive strengths of this system indicated the need to concentrate the balance of the work in this phase on the dense-graded Type IVb specification.

Because of the lower-than-expected air void content being generated some concern developed over the validity of the Rice specific gravity (ASTM D-2041-71) test for these types of materials. Therefore, the specific gravity of mixes using Gradation No.'s 1 through 4 was rechecked using a gravimetric method. The results indicated virtually no difference between the two test procedures.

Mix properties using Gradation No's. 1 and 2 provided the best combination of strength and air void content. Gradation No. 3 was prepared using about 18 percent fly ash in place of the fines fraction. Preliminary tests on these mixes indicated good strength but low air voids. The potential for studying fly ash-aggregate mixtures of sulfur concrete warrants further investigation, since this activity was considered outside the scope of this project.

3.2 Phase II - Material Characterization and Structural Analysis

3.2.1 Materials Characterization. The five sulfur concrete mix designs listed in Table 1 were subjected to the following series of tests in accordance with the plan shown in Figure 4:

Rice Specific Gravity	ASTM D2041
Unconfined Compression	ASTM C684
Freeze-thaw Durability	ASTM C666
Flexural Fatigue	VESYS Manual [7]
Repeated Load Creep	VESYS Manual [7]

The results of the specific gravity and compression tests are shown in Table 5.

Table 5. Results of Specific Gravity and Compression Tests on Sulfur Concrete Specimens.

Sample Number	Plasticizer Content (%)	O/DCPD Ratio	Binder in Mix (%)	Specific Gravity	Compression Strength psi
M92	5	50/50	11	2.41	4600
M93	10	60/40	11	2.35	4200
M94	20	75/25	11	2.26	1700
M95	30	75/25	9	2.29	900
M96	40	75/25	9	2.32	600

1 psi = 6.9 kPa

Specific Gravity tends to decrease as more plasticizer is added to the system. This would be expected since the specific gravity of sulfur to modifiers is about 2 to 1. It would appear that the compressive strength drops significantly as the plasticizer content exceeds 20 percent. Reducing the binder content from 11 to 9 percent did not offset the loss of strength due to the high degree of plasticization represented by mixes M95 and 96.

Freeze-thaw. Freeze-thaw tests were run in accordance with ASTM C666. This test provides for the alternate freezing and thawing of 3 in. (7.6 cm) x 3 in. (7.6 cm) x 15 in. (38.1 cm) specimens by cycling their temperature between 40°F (4.4°C) and 0°F (-18°C) at a rate of 6 cycles/day. Failures are associated with loss of stiffness and/or cracking of the specimens. The relative dynamic modulus E_c which is a measure of the reduction in strength is calculated by

$$E_c = \left(\frac{\eta_c}{\eta_o} \right)^2 \times 100\% \quad (8)$$

where E_c = relative dynamic modulus (%) after c cycles of freezing and thawing,

n_0 = fundamental transverse frequency before testing (0 cycles)

n_c = fundamental transverse frequency after c cycles

In this test the specimen is subjected to a forced vibration of varying frequencies. The frequency which produces a maximum indication with a well defined peak is recorded as n_c . This parameter is determined periodically during the course of the test. When the calculated value of E_c drops below 60%, the test is concluded.

The results of the freeze-thaw durability tests are given in Table 6. These tests were run in accordance with ASTM C-666 using 3 in. (7.2 cm) by 3 in. (7.2 cm) and 15 in. (38.1 cm) long samples. This procedure is used primarily with rigid concretes such as PCC. In the absence of a standardized procedure for flexible systems this procedure has also been used in a limited sense for qualitative evaluations of asphaltic concretes. In the strict sense, therefore, only the 5, 10, 20 and 30 percent plasticized systems would qualify for this test since their compressive strengths (see Table 5) were in the vicinity of 10^6 psi (6.9×10^6 kPa). The 40 percent plasticized system would most likely qualify as a "flexible" concrete.

The durability of a material subject to ASTM C-666 is established as the number of cycles at which the Relative Dynamic Modulus, E_c , is reduced to 60 percent of its original value. On this basis, the 40 percent plasticized (flexible) system would have lasted about 30 cycles and the others would have failed somewhere between 35 and 69 cycles. Of the four systems considered to be rigid (M92, 93, 94 and 95) the 5 percent system indicated the poorest durability. For rigid systems such as PCC as much as 300 cycles are normally achieved. Based on TTI's experience with dense-graded asphaltic concretes, durabilities of at least 75 to 100 cycles can be expected.

The poor performance of the 5 percent system is not consistent with earlier results reported by the Bureau [6] which indicated good durability out to 300 cycles. One possible cause of

this performance was the poor surface quality of the specimens. There were a number of surface defects and debonds between aggregate and binder noticed during the test. Based on the results shown in Table 8 the freeze-thaw durability of these mixtures cannot be established without additional testing.

Table 6. Results of Freeze-thaw Tests in Sulfur Concrete Mixtures.

BOM Mixture Number	Plasticizer Content %	Relative Dynamic Modulus, E_c , %		
		Number of Cycles		
		0	35	69
M92	5	100	70	29
M93	10	100	76	52
M94	20	100	90	33
M95	30	100	72	49
M96	40	100	51	14

Flexural Fatigue. The fatigue behavior of five sulfur concrete mixtures was evaluated by means of flexural beam fatigue testing. Third point, upward loading stress-controlled tests were performed in strict accordance with the VESYS IIM Users Manual [7]. Since strains continually increased throughout the duration of this test, the strain recorded was the stress amplitude divided by the resilient modulus. The beams were tested in a haversine loading mode at a frequency of 100 cycles per minute.

The fatigue response to each of the mixes can be initially compared by reviewing Figure 7. Fatigue curves for mixes M92, 93, 94, 95 and 96 are plotted together with the fatigue curve of a laboratory standard asphaltic concrete mixture. The latter utilized an AC-10 asphalt cement and a densely graded crushed limestone aggregate.

The expression of the phenomenological fatigue law most often used to express these data is given by Equation 4. The

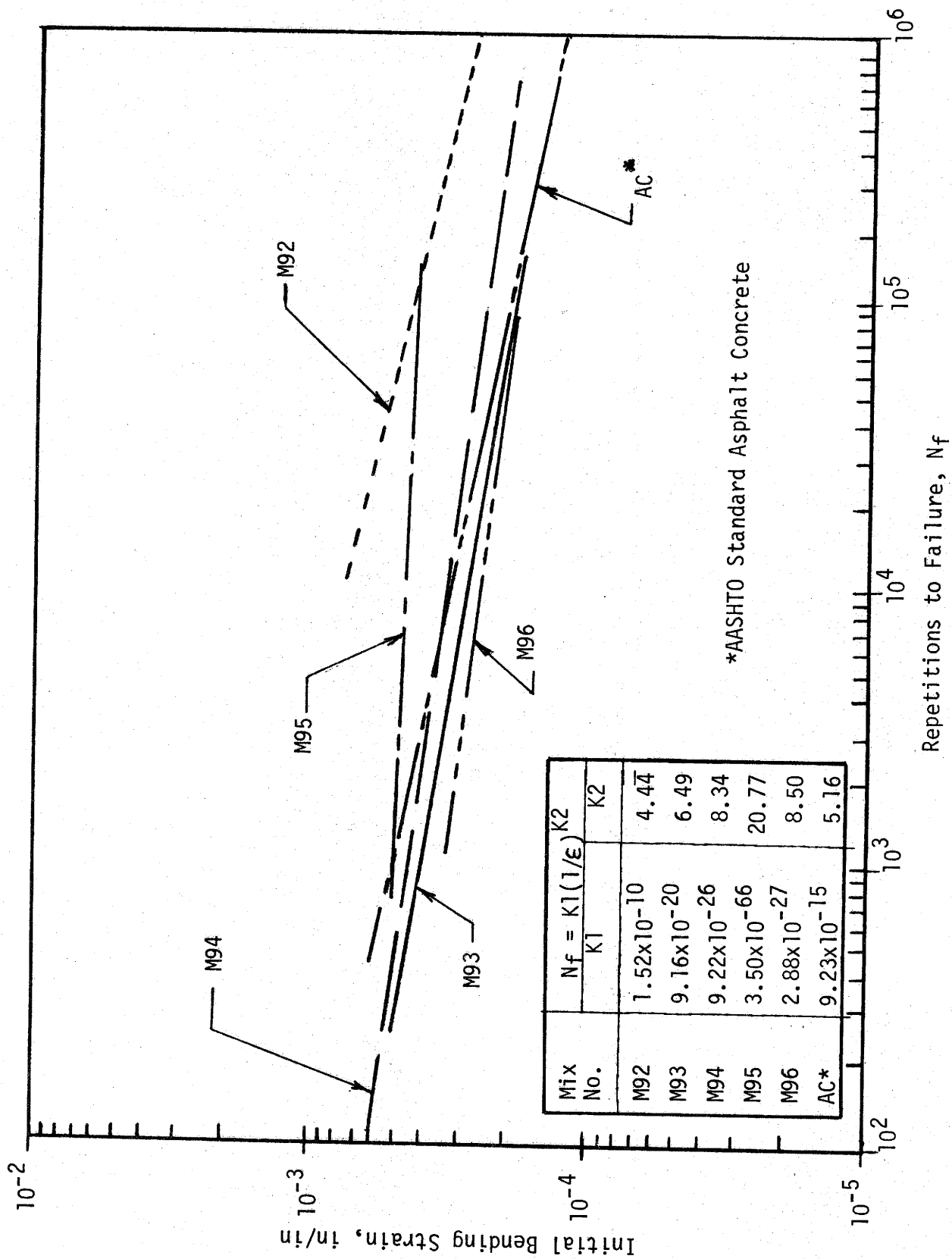


Figure 7. Initial Bending Strain vs Load Repetitions to Failure for Sulphur Concrete Mixes

equation attached to each fatigue curve is of this form. Note, particularly, the values of the slope K2 which, together with a visual inspection of the slopes of the fatigue curves, are a clear indication of the relative fatigue response of these materials. It should be noted that the slopes are significantly flatter than those typical of the asphalt concrete. It should be noted that the slope of K2, of the laboratory control mix is 5.00 (a slope of between 2.8 and 5.0 is typical) [11,12], which is flatter than is often observed.

Next it is interesting to evaluate the fatigue curves of the mixtures individually. Mixture M92 is most like asphalt concrete in terms of its fatigue slope. However, the relatively very large K1 (1.52×10^{-10}) is indicative of a fatigue behavior quite superior to asphalt concrete, at least in the controlled-stress environment. A controlled-strain mode of testing could yield quite different results.

Mixtures M94, 95 and 96 all possess very flat slopes of 8.34, 20.77 and 8.50, respectively. These slopes indicate the existence of a fatigue limit similar to that which one would expect in portland cement concrete. It is suggested that a relationship between stress ratio (applied stress to maximum allowable stress) and number of load applications to failure might be a more meaningful index by which to evaluate such data. The maximum allowable stress, in this case, could be obtained by means of a flexural strength test of a specimen beam. The modulus of rupture as determined for portland cement concrete beams is such a property.

The slope of the fatigue curve for mixture M93 (6.49) was intermediate between the typical asphalt concrete (5.00) and the relatively flat slopes of the other mixtures.

These fatigue relationships were used as input to the VESYS IIM structural model and will be discussed in a following section. The fatigue cracking model used in VESYS is based on Miner's law:

$$D_i = \sum_{i=1}^j \frac{n_i}{N_i} \quad (9)$$

where:

n_i = number of load cycles during the i to time interval.

N_i = number of cycles to failure under temperature and strain condition of the i to time interval.

$$D_i = \text{Damage Index} = K1_i \left(\frac{1}{E_i} \right)^{K2_i}$$

When the damage index is equal to or greater than unity, cracking is considered to occur. The area of cracking or extent of cracking which propagates following the initial cracks is interpreted probabilistically, based on the mean and variance of $K1_i$, $K2_i$ and E_i , all considered to be random variables. Taking the necessary moment about the mean using a Taylor's expansion, the expected values, variance and probabilities of failure are evaluated.

In order to obtain a realistic estimate of fatigue failure it is necessary to not only identify the laboratory flexural fatigue values $K1$ and $K2$, but also establish the laboratory-to-field shift factor. The shift has been found in the literature to range from 3 to 200 [11, 13]. The need to introduce such a factor is due to the differences in boundary conditions between the laboratory and field, differences in rest periods between loadings, residual stresses induced in the field but not in the laboratory-measured visco-elastic material characteristics, etc. These shift factors have also been shown to be a function of temperature [14].

VESYS does not include a laboratory-to-field shift mechanism and it would be difficult to indiscriminately appropriate one. Thus, in this analysis no attempt was made to shift the fatigue curve. The result is an unrealistically rapid failure by cracking with all mixtures, sulfur concrete as well as asphalt concrete. However, the cracking index is still valid as a comparative index. The difficulty in establishing the field shift factor is not necessarily a shortcoming of the VESYS analysis, but of the VESYS procedure.

The comparison between the sulfur concrete mixes and asphalt concrete was made because of the extensive use of asphalt concrete as a paving material and because of the proliferation of fatigue data on asphalt concrete. However, it may be just as likely that sulfur concrete will compete with portland cement concrete (PCC) as a paving material. In this case, we are also obligated to compare the fatigue test results with those of PCC.

Portland cement concrete (PCC) fatigue data are normally presented as stress ratio, S , versus number of applications at that ratio to failure, where S is defined as the ratio of actual induced stress to the rupture modulus. These plots are called S-N curves. Figure 8 is an S-N curve comparing the fatigue data for the sulfur concrete mixtures with similar data for portland cement concrete as well as other sulfur concrete fatigue data reported in the literature. The sulfur concrete represented by the data of Lee and Klaiber [15] was prepared using Chement 2000, a 5 percent plasticized rigid binder manufactured by Chemical Enterprises of Odessa, Texas, using the Bureau's mixed-modifier process.

Unfortunately, insufficient data are available for a suitable presentation of the S-N curves in this comparative format. More testing is necessary together with reliable determination of the rupture modulus of the sulfur concrete beam. However, certain trends can be identified. First, the rupture moduli for mixes M92 (5 percent plasticizer) and M93 (10 percent plasticizer) were recorded as 815 and 744 psi, respectively. The plots on Figure 8 reflect the fatigue results for these low plasticizer mixes. The fatigue responses are inferior to PCC in both cases. The compressive strengths and rupture moduli, on the other hand, are similar to PCC.

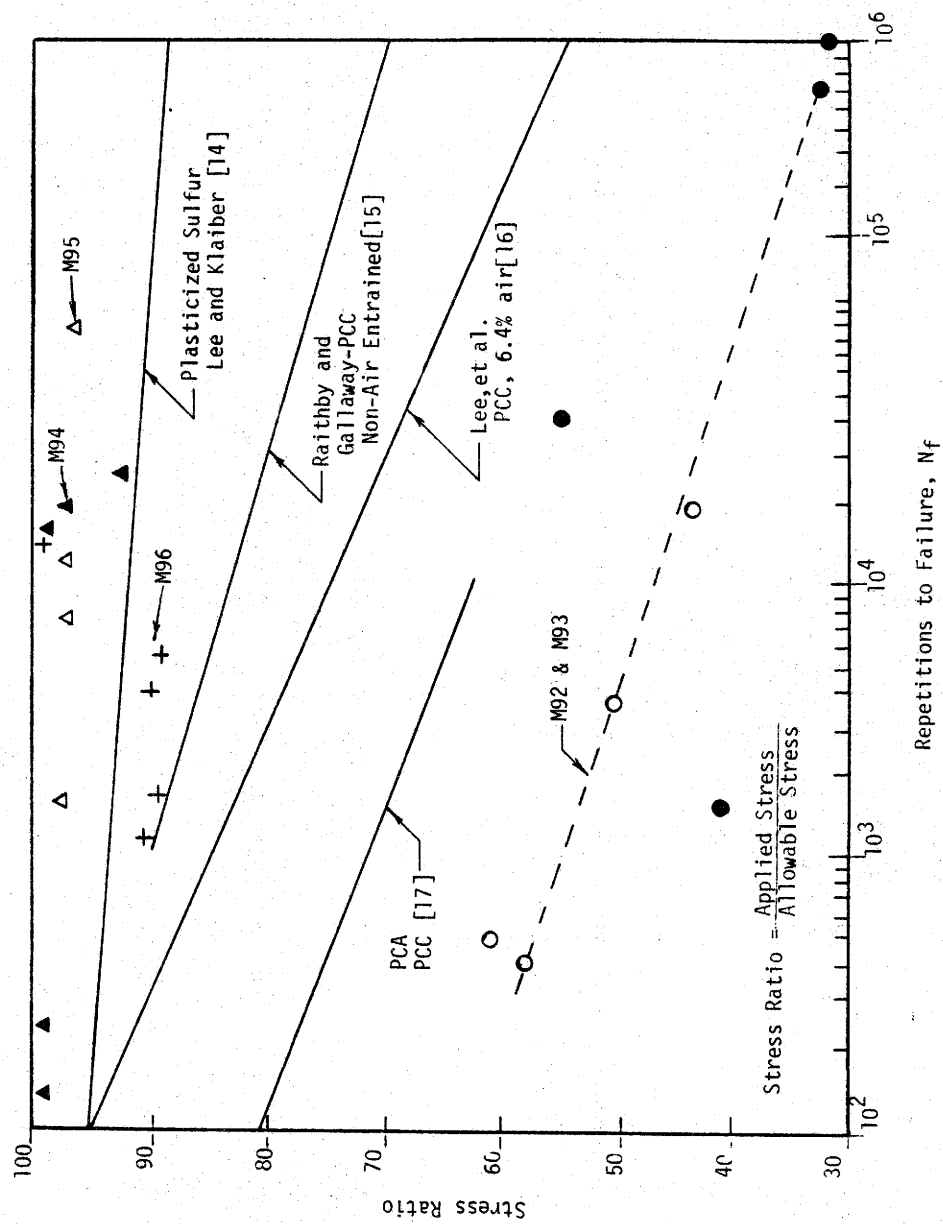


Figure 8. Comparative S-N Curves for Sulfur Concrete and Portland Cement Concrete.

With the addition of plasticizer (20% for M94, 30% for M95 and 40% for M96), the compressive strengths were substantially reduced (see Table 5). It is likely that the rupture moduli are reduced also. These rupture moduli were not determined and cannot be inferred from the available data. It is clear, however, that the fatigue life (as a function of stress ratio) is substantially increased by a plasticizer content of twenty percent and above. This is inferred from the existing fatigue data because the high stress levels induced in the fatigue tests of mixes 94, 95 and 96 produced favorable fatigue results. The high flexural stresses induced in the fatigue beams produced a very high stress ratio for the beam in question.

More fatigue testing, both of the controlled stress and controlled strain variety, is necessary in order to establish reliable relationships. An extended fatigue testing program is recommended based on these data which illustrate the significant role of the plasticizer to sulfur ratio (P/S) on the fatigue response.

Repeated-Load Creep. All creep testing was performed in strict accordance with the procedure detailed in the VESYS Users Manual [7]. Figure 9 presents the creep results obtained for the four sulfur concrete mixtures and the control mixture evaluated in this study. These creep curves were developed from 77°F (25°C) data.

The sulfur concrete mixtures were all significantly stiffer than the asphalt concrete control mixture. In addition, the sulfur concrete mixtures, with the exception of mixture M96, all exhibited near elastic behavior in terms of their response to the duration of applied load.

The elastic response of the sulfur concrete mixtures is further illustrated by the BETA factor and time-temperature shift factor a_t . The parameter BETA, β , is defined as the ratio of $\log a_t$ to the temperature differential over which a_t is calculated. For the sulfur concrete and control mixtures, the computed β factors are presented in Table 7. The zero values for β for mixtures M92, 93, 94 and 95 clearly indicate that these materials respond essentially elastically. Mixture M96 has a β factor and a_t much smaller than the control.

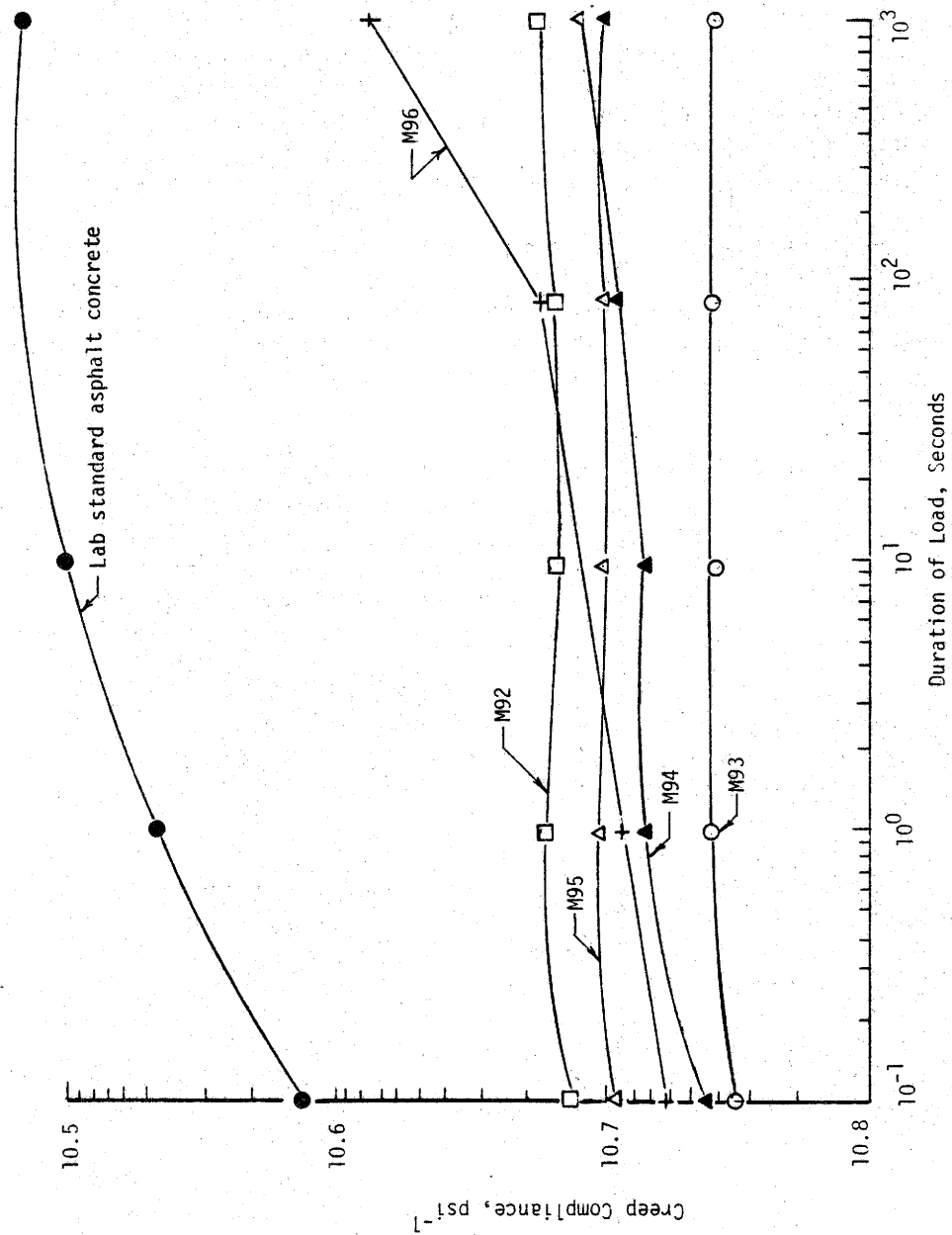


Figure 9. Creep Curves for Sulphur Concrete Mixtures and Laboratory Standard Asphaltic Concrete.

Table 7. Summary of Stiffness Moduli and Beta Values.

Mixture Identification	Stiffness Modulus,* psi	Beta (B) Value $B = \frac{\log A_T}{(T_o - T)}$
Laboratory Standard AC-10 and Crushed Limestone	7.14×10^5	0.087
Mixture 92	7.7×10^6	0
Mixture 93	3.0×10^7	0
Mixture 94	2.5×10^7	0
Mixture 95	1.11×10^7	0
Mixture 96	1.67×10^7	0.037

* at 10^{-1} sec load duration

Permanent deformation test results are summarized in Figure 10 and in Table 8. Basically alpha, α , values of between 0.63 and 0.83 are considered typical for asphalt concrete [11, 12]. The unusually low values for sulfur concrete mixtures M92 and M93 occur due to the significant increase in deformation resulting from the longer loading duration. It is impossible, at this time, to explain the reason for the rapid increases in deformation under longer loading times for mixtures M92 and M96 based on these limited data.

GNU is a very difficult parameter to ascribe a physical meaning. However, the extremely low values of GNU shown in Table 8 generally indicate a very low potential to rut.

3.2.2 Structural Analysis. The VESYS IIM Structural subsystem was used to evaluate the response of the sulfur concrete mixtures M92, 93, 94, 95 as structural layers in a pavement system.

A conventional pavement structure was selected with a soft, clay subgrade and a 10 in. (254 mm) high quality crushed stone aggregate base. Two thicknesses of surface courses were placed on top: a two-inch surface (thin) and a six-inch surface (thick). Three climatic regions were evaluated: a cold climate, a moderate climate and a hot climate using the temperatures shown in Table 3.

In each analysis performance over a 20-year service period was evaluated, and traffic was assumed to have grown from 3,000 to 9,000 vehicles per day. Overall, the sulfur concrete pavements performed very well when compared to the asphalt concrete control mixture. This is illustrated in Figures 11 and 12 by plotting the change in present serviceability index, (PSI), with time for the thick and thin pavements, respectively.

These results, however, must be analyzed with some degree of caution. The formulas for computing present serviceability index in VESYS IIM are very heavily weighted toward rut depth and slope variance. Both these parameters would be expected to be very good for the essentially-elastic, stiff sulfur concrete.

It is interesting to note the similarity between the performance curves of the control and sulfur concrete mixture M92 for both thick and thin pavement sections. A similarity was also noted between mixture M92 and the control in terms of the beam fatigue data and the permanent deformation curves.

Table 8. Summary of Permanent Deformation Parameters, Alpha and GNu.

Mixture Tested	Alpha, α	GNu, μ
Laboratory Standard: AC-10 and Crushed Limestone	0.663	0.115
Mixture 92	0.100	0.0003
Mixture 93	0.800	0.0505
Mixture 94	0.810	0.0672
Mixture 96	0.2800	0.0097

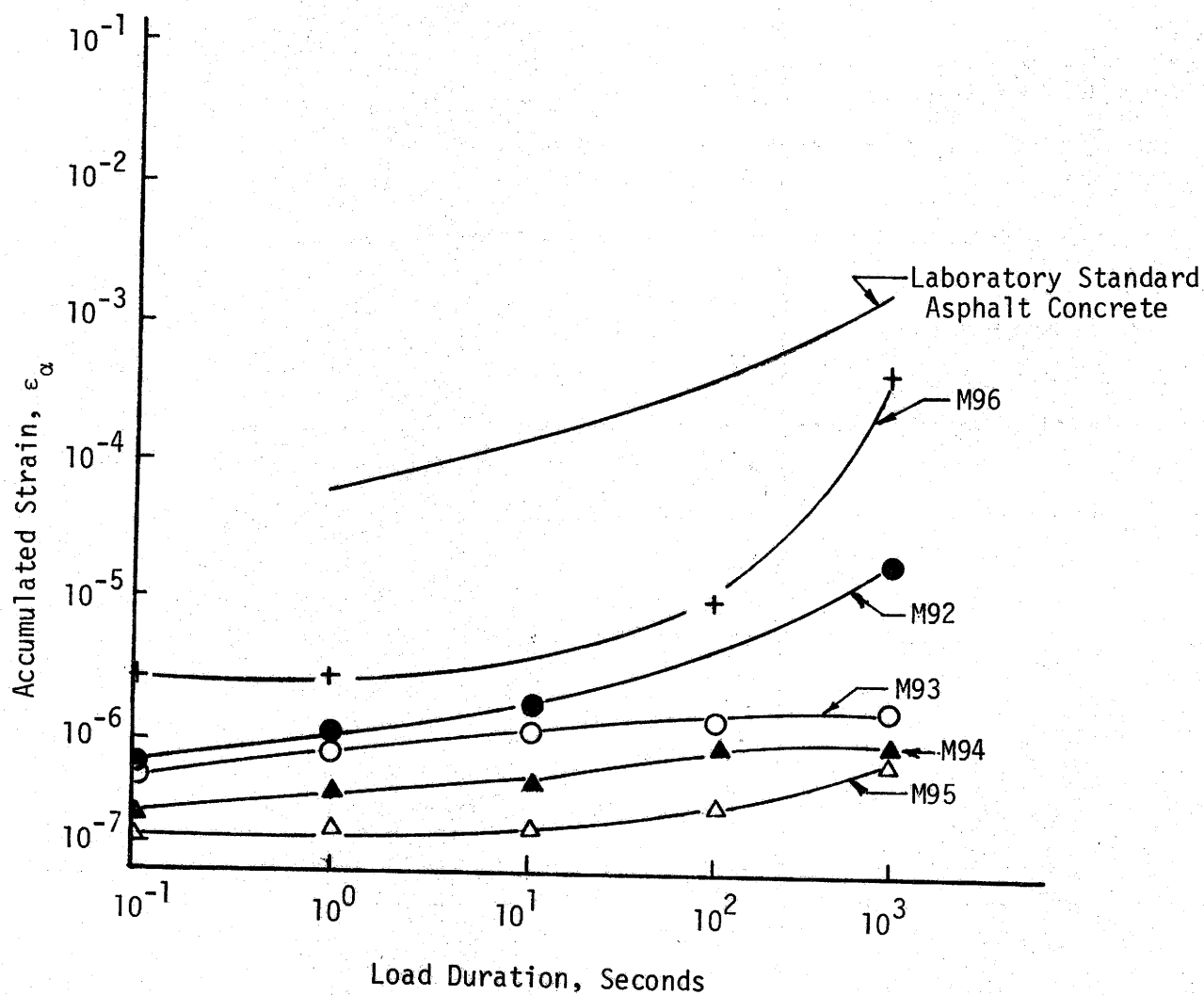


Figure 10. Permanent Deformation versus Load Duration for Sulfur Concrete Mixtures and Laboratory Standard Asphaltic Concrete.

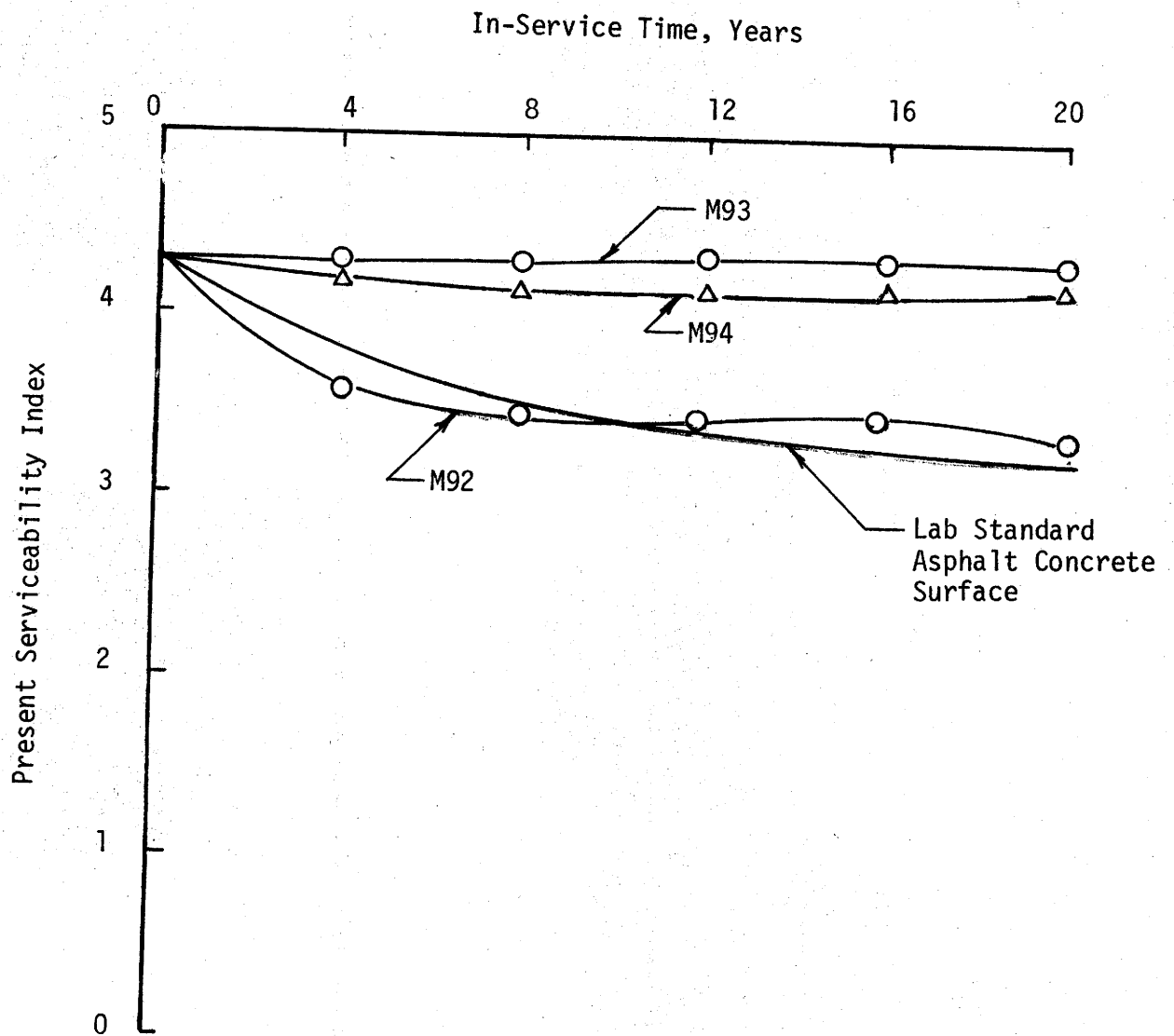


Figure 11. Present Serviceability Loss with Time for Thin Pavement Surfaces (2 Inches) for Sulfur-Concrete and Laboratory Standards Asphaltic Concrete.

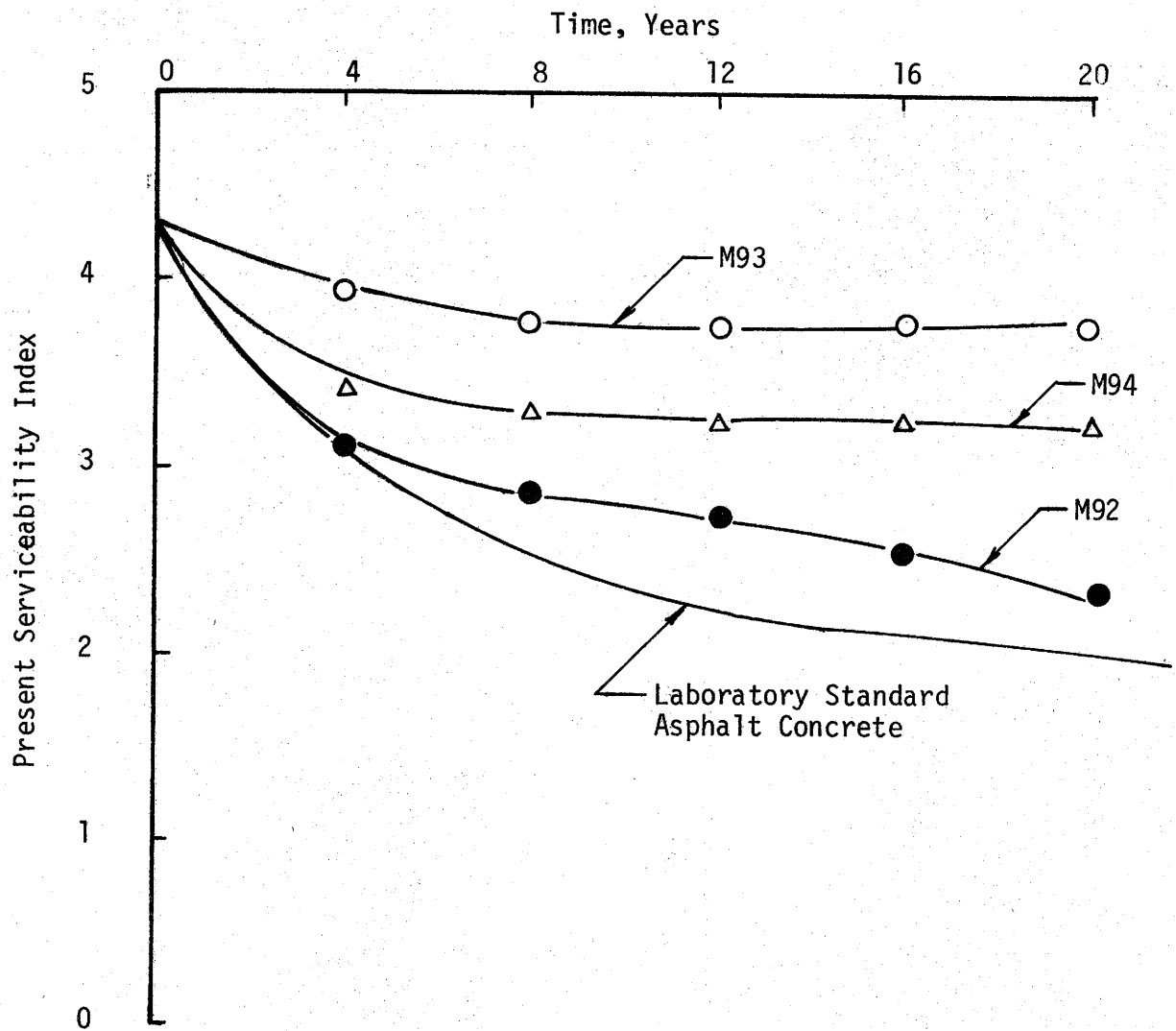


Figure 12. Present Serviceability Loss With Time for Thick Surfaces (6 Inches) for Sulfur Concrete and Laboratory Standard Asphaltic Concrete.

The slope variance is defined as the variation of deformation along the longitudinal profile of the roadway. The variation is assumed to be the result of variability in material properties, construction practice and the variation in the local-deflection response. The inordinately high slope variance for mixture M92 as shown in Figure 13 is due largely to the variability of the material properties obtained in creep compliance and permanent deformation characterization tests.

Figures 14 and 15 plot the damage cracking index versus years of pavement service for thick and thin pavements, respectively. These results are indicative of a controlled-stress mode of fatigue testing. Thus, the results should be valid for the thick, 6 in. (15.2 cm) section and for the thin, 2 in. 5.1 cm) if it rests over a very stiff subgrade or thick, stiff base. A controlled-strain mode of flexural fatigue must be used to verify the ability of the sulfur concrete to perform in thin layers over weak subgrades. This type testing was not performed in this program.

Rut depth is summarized in Figure 16. Clearly, the rutting potential of the sulfur concrete mixtures is not a problem in any normal pavement system.

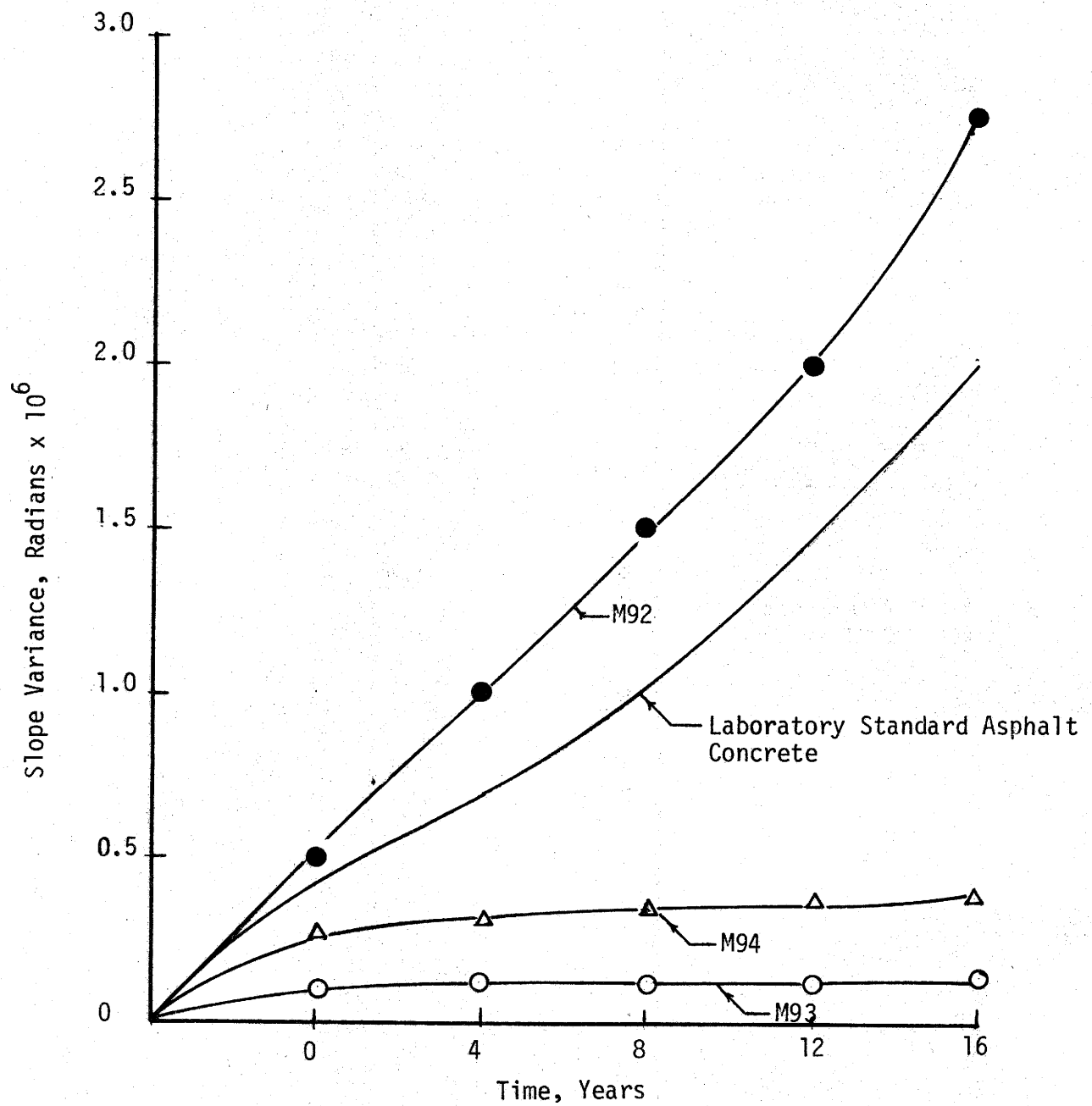


Figure 13. Change in Slope Variance (Roughness) With Time (Age) for Sulfur Concrete and Laboratory Standard Asphaltic Concrete.

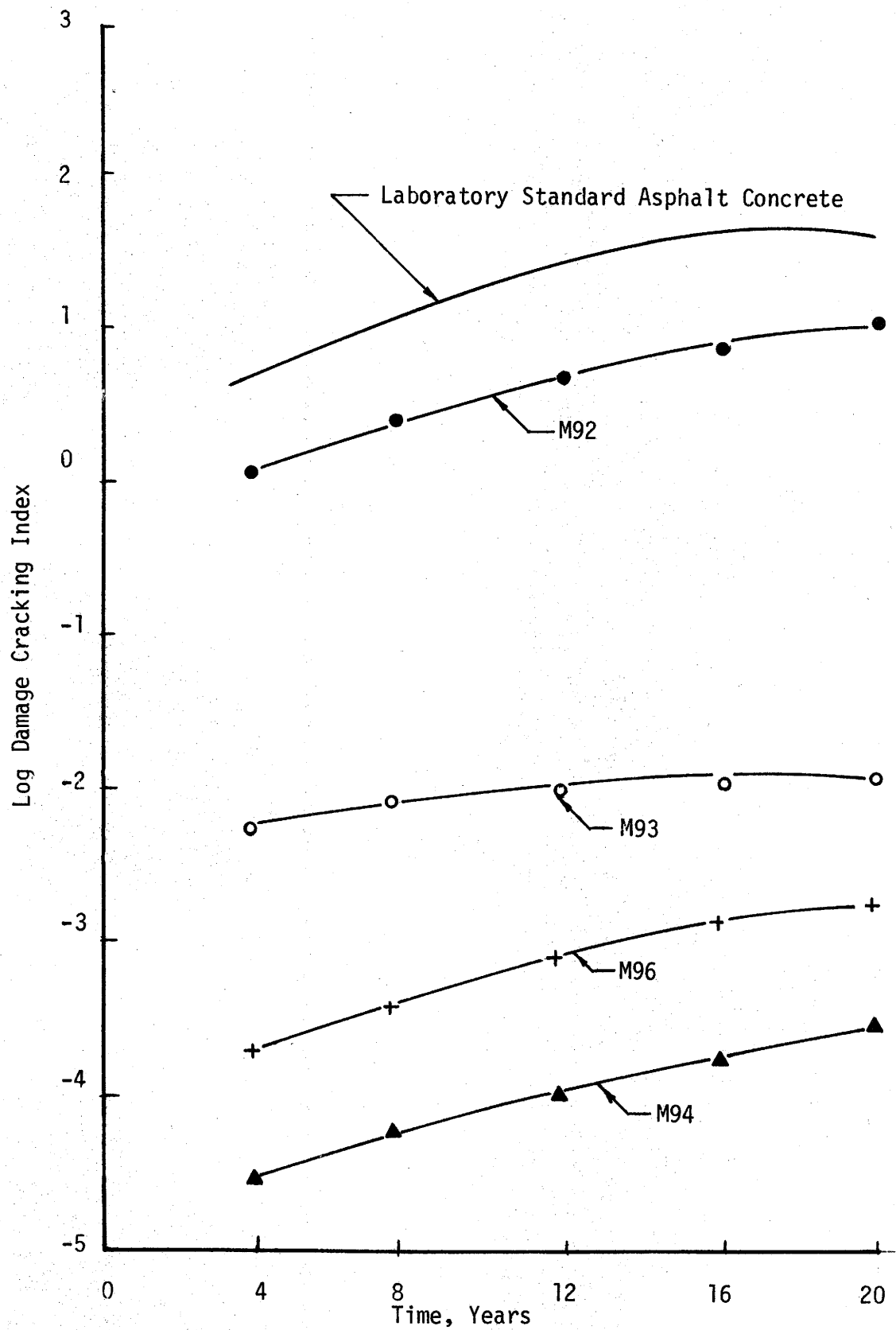


Figure 14. Increase in Cracking Damage Index With Times for Thin Pavements of Sulfur Concrete and Laboratory Standard Asphaltic Concrete.

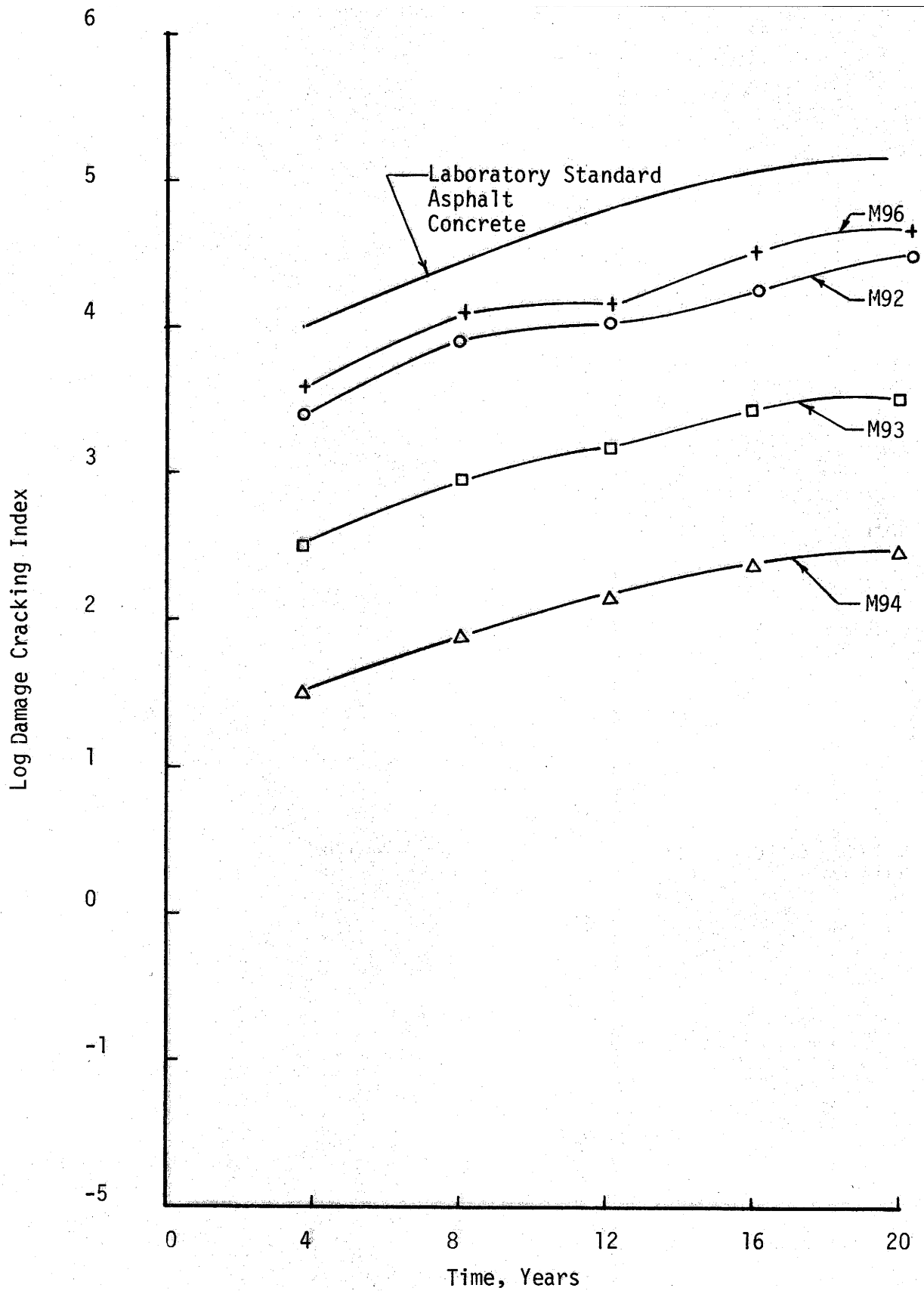


Figure 15. Increase in Cracking Damage Index with Time for Thick Pavements for Sulfur Concrete and Laboratory Standard Asphaltic Concrete.

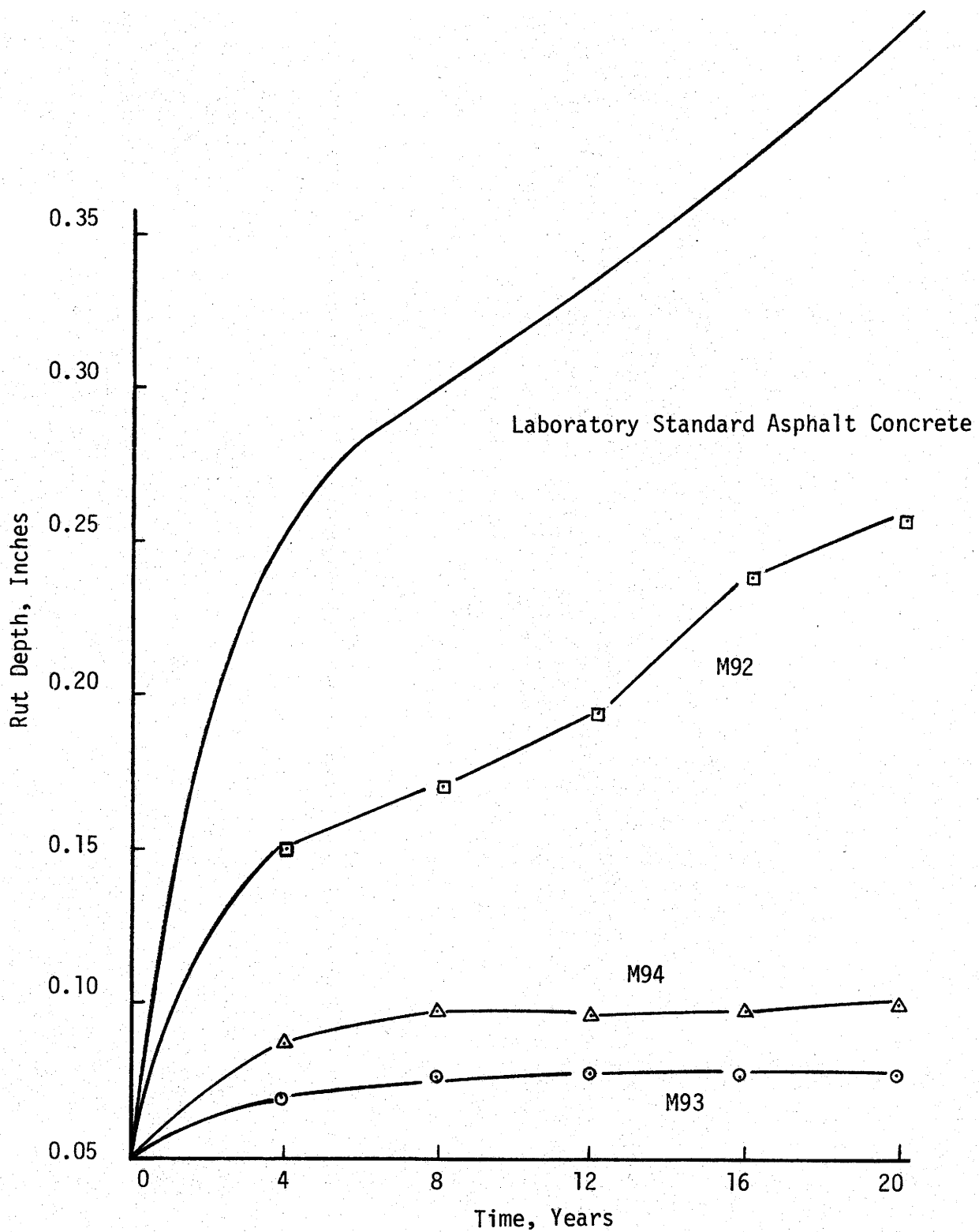


Figure 16. Increase in Rut Depth with Age of Sulfur Concrete and Laboratory Standard Asphaltic Concrete.

3.3 Phase III - Duomorph Testing and Results. To provide a data base to compare with the Duomorph results, the five binders furnished by the Bureau (see Section 2.1) were tested for resilient modulus at three different temperatures: 69, 90 and 134°F (20, 32 and 56°C). The results of these tests are given in Table 9.

Table 9. Resilient Modulus versus Temperature and Degree of Plasticization of Unfilled Binder.

BOM Mixture Number	Plasticizer Content %	Resilient Modulus*, psi x 10 ⁻⁶ Test Temperature °F (°C)		
		68 (20)	90 (32)	134 (56)
M92	5	0.967	0.580	0.342
M93	10	0.652	0.350	0.196
M94	20	0.126	.059	0.047
M95	30	0.104	.048	0.031
M96	40	0.014	.007	Too soft

*Poisson's ratio used in these calculations was 0.35

The above data represent the average of three tests. The moduli were obtained using a stress of 20 psi applied over 0.1 sec. The 40 percent binder was too soft to test at the maximum temperature. During loading the sample would develop flat spots and experience sufficient deformation to invalidate the test results.

3.3.1 Sample Preparation Problems. At the outset it was planned that the Duomorph be used as a surface gage. In this testing mode, changes in surface properties due to exposure to different storage conditions would be more pronounced. The analysis for the Duomorph acting on a semi-infinite slab already existed [8]. Following calibration of the Duomorph in air (i.e. unbonded) some preliminary tests were run to obtain a frequency which would provide results consistent with the rate of

loading in the resilient modulus test. The Duomorph frequency was established at 10 cps which corresponds to the loading rate of the resilient modulus test which induces a 75 lb (34 kg.) in 0.1 sec. It became apparent that the surface of the binders did not couple well with the Duomorph giving results not too different from those generated in air. One approach to resolving this problem was to add some weight to the Duomorph to improve the contact with the binder surface. Up to 75 grams were necessary to effect reasonable interfacial contact, however, this weight violated the assumption of semi-infiniteness of the analysis. After numerous attempts to resolve this problem some of which involved the application of pressure to a low modulus silicone rubber sheet placed over the Duomorph to distribute the load, it was decided to go to an embedded transducer testing mode.

Each binder was heated above the melting point in 150 ml beakers and the Duomorph was submerged below the surface in a horizontal orientation. After a few seconds of activation, the response decayed rapidly indicating a debond or breakdown in the contact between the transducer and the binder. This is the first time this type of performance was experienced in a Duomorph test.

As a further check on the quality of the interfacial bond a 10 gram sample of binder was heated above the melting point and then poured over a portland cement concrete surface and allowed to cool. When the binder reached room temperature it required only a slight amount of shearing force to uncouple the binder from the concrete surface. It was felt that the high-rate shearing action of the Duomorph in the binder could cause a similar interfacial uncoupling and produce results similar to that of an unbonded transducer.

In another attempt to affect a test procedure compatible with the plasticized sulfur binders, it was decided to prepare samples in thin circular discs about the same size as the Duomorph (i.e. 0.75 in. [1.91 cm] diameter). These samples were molded into 0.03 in. (0.8 cm) thick discs. The discs were placed in a cavity of a 4 in. (10.2 cm) square by 0.06 in. (0.16 cm) thick fiber glass sheet as shown in Figure 17. The Duomorph was placed on top of the disc and a thin latex sheet subsequently placed over the Duomorph. This assembly was sandwiched in a

0.25 in (0.62 cm) thick frame with a 3 in. (7.6 cm) square opening. Finally, each end was covered with a metal plate fabricated with a fitting for pressurizing the cavity. The various segments of this fixture are shown in Figures 17 through 20. The latter shows the complete assembly with hook-up to a N₂ bottle and readout equipment. All data to be discussed below were generated with this test setup.

3.3.2 Test Results. Two types of tests were conducted on the five binders:

1. Modulus versus Temperature; $68^{\circ}\text{F} \leq T \leq 250^{\circ}\text{F}$ ($25^{\circ}\text{C} \leq T \leq 121^{\circ}\text{C}$)
2. Modulus versus Temperature and Frequency; 10 Hz and 100 Hz

The temperature range was selected to provide data over the liquid to solid transition phase of the binder. The frequency range was selected to establish the extent, if any, of rate-dependence (viscoelastic) characteristics inherent in these binders. The results of the two types of tests are shown in Figures 21 and 22.

Figure 21 shows a comparison of moduli measured by the Duomorph with those generated in the resilient modulus tests (Table 10). Except for the 40 percent plasticized binder the resilient moduli at 68°F (25°C) compared closely with that measured with the Duomorph. However, significant reductions in stiffness appear as the temperature increased. The Duomorph indicated a relatively constant stiffness up to the softening point; 150°F (66°C) of the binders. Duomorph stiffnesses show slight changes with temperature in the 20 percent plasticized binder and tend to increase with plasticizer content.

Similar behavior was indicated when the excitation frequency for the Duomorph was increased from 10 to 100 Hz as shown in Figure 22. An increase in load rate on a viscoelastic material should produce an increase in stiffness. Only the 40 percent plasticized binder incurred an increase of any significance.

No reason can be given for the apparent difference in temperature (rate) dependence reflected by the two test methods shown in Figure 21. It should be noted that the Duomorph

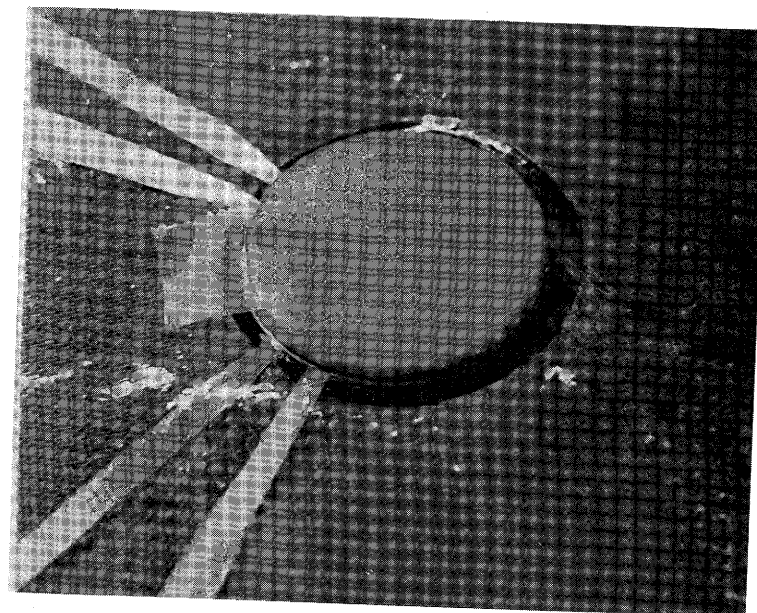


Figure 17. Test Sample in Fiberglass
Cavity Containing Duomorph.

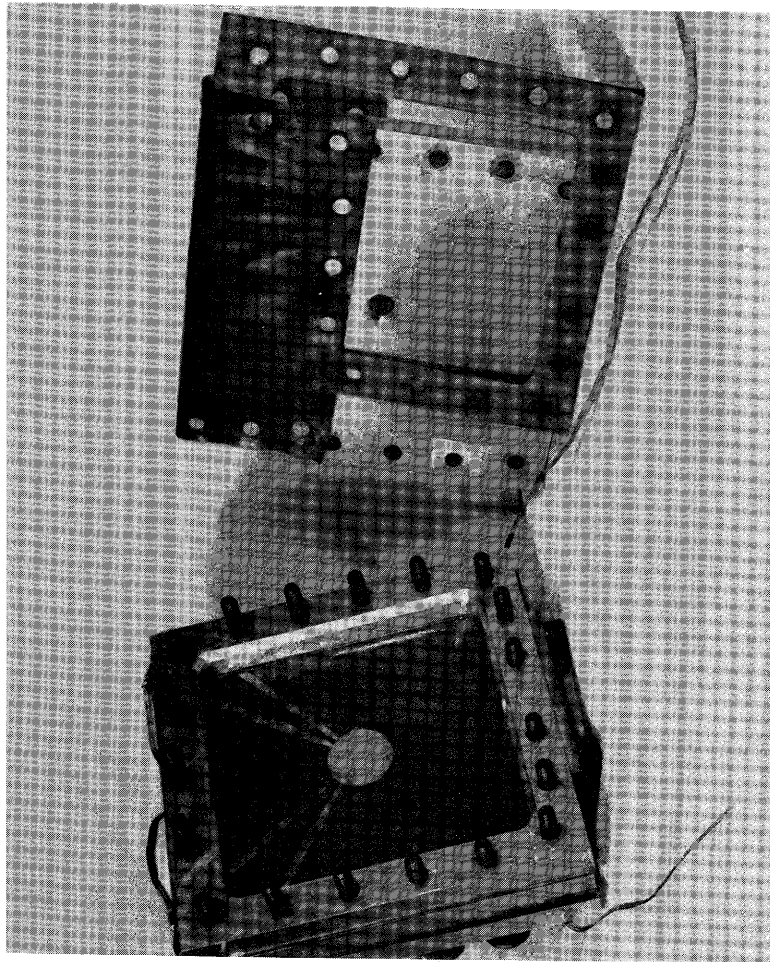


Figure 18. Internal view of Duomorph Test Fixture
and Components.

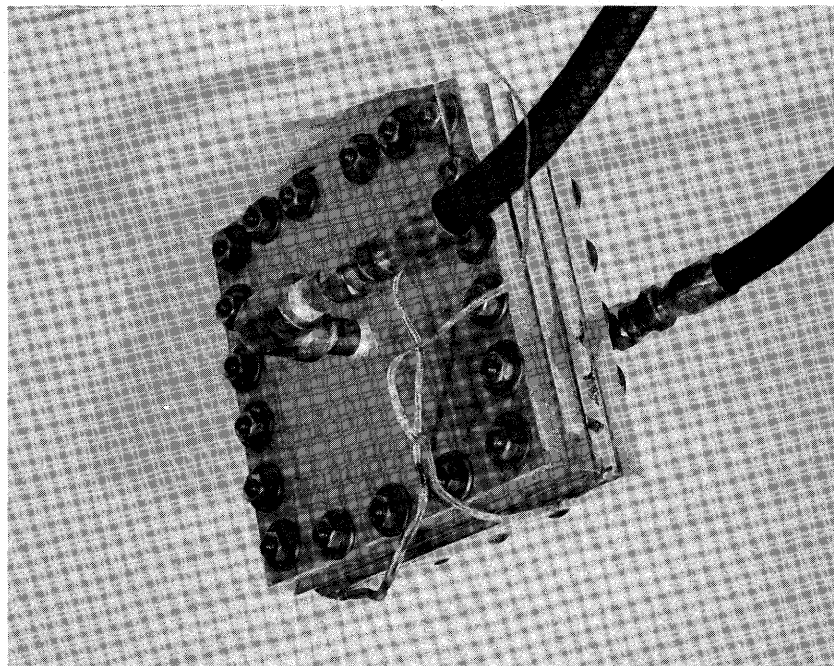


Figure 19. Duomorph Test Fixture.

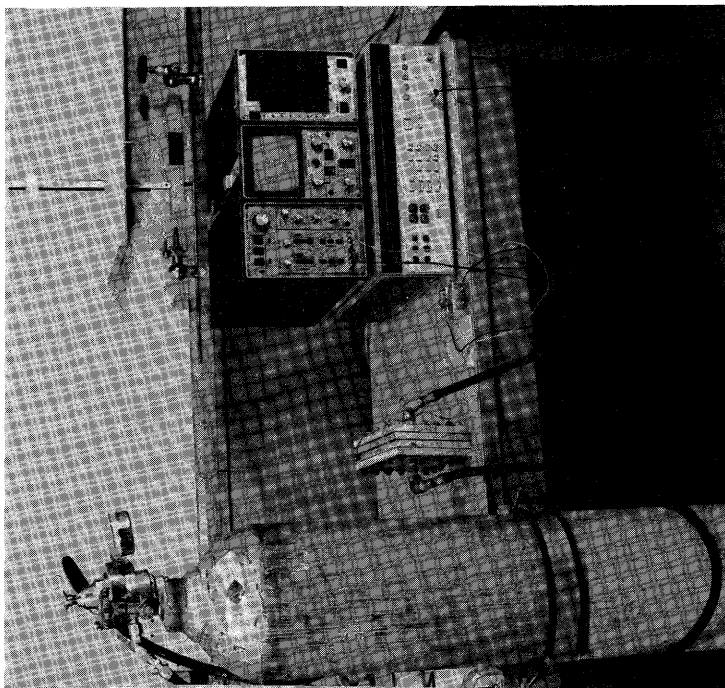


Figure 20. Duomorph Test Set Up with N₂ Bottle and Readout System.

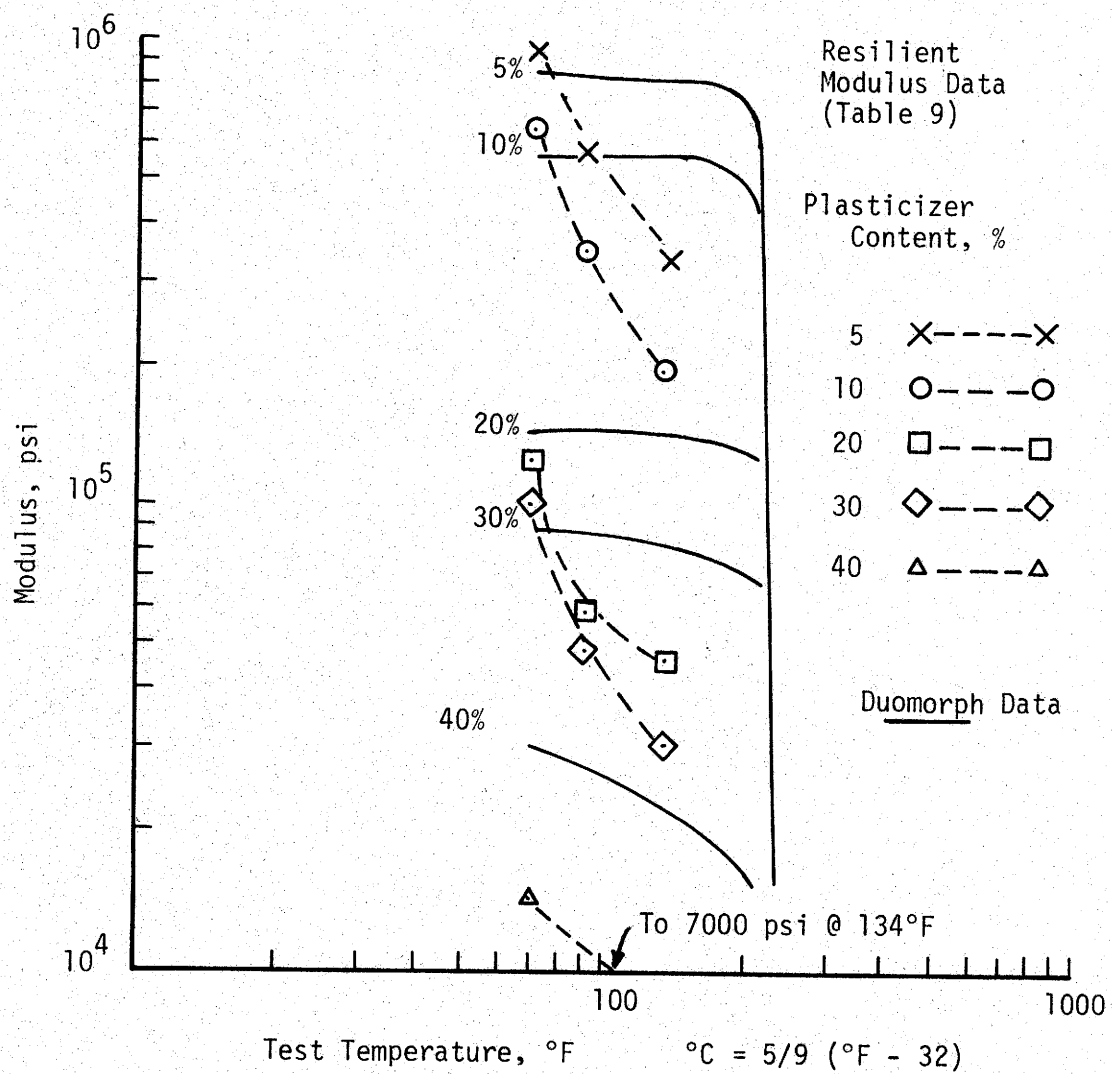


Figure 21. Comparison of Modulus versus Temperature from Duomorph and Resilient Modulus Tests at Various Plasticized Sulfur P/S Ratios.

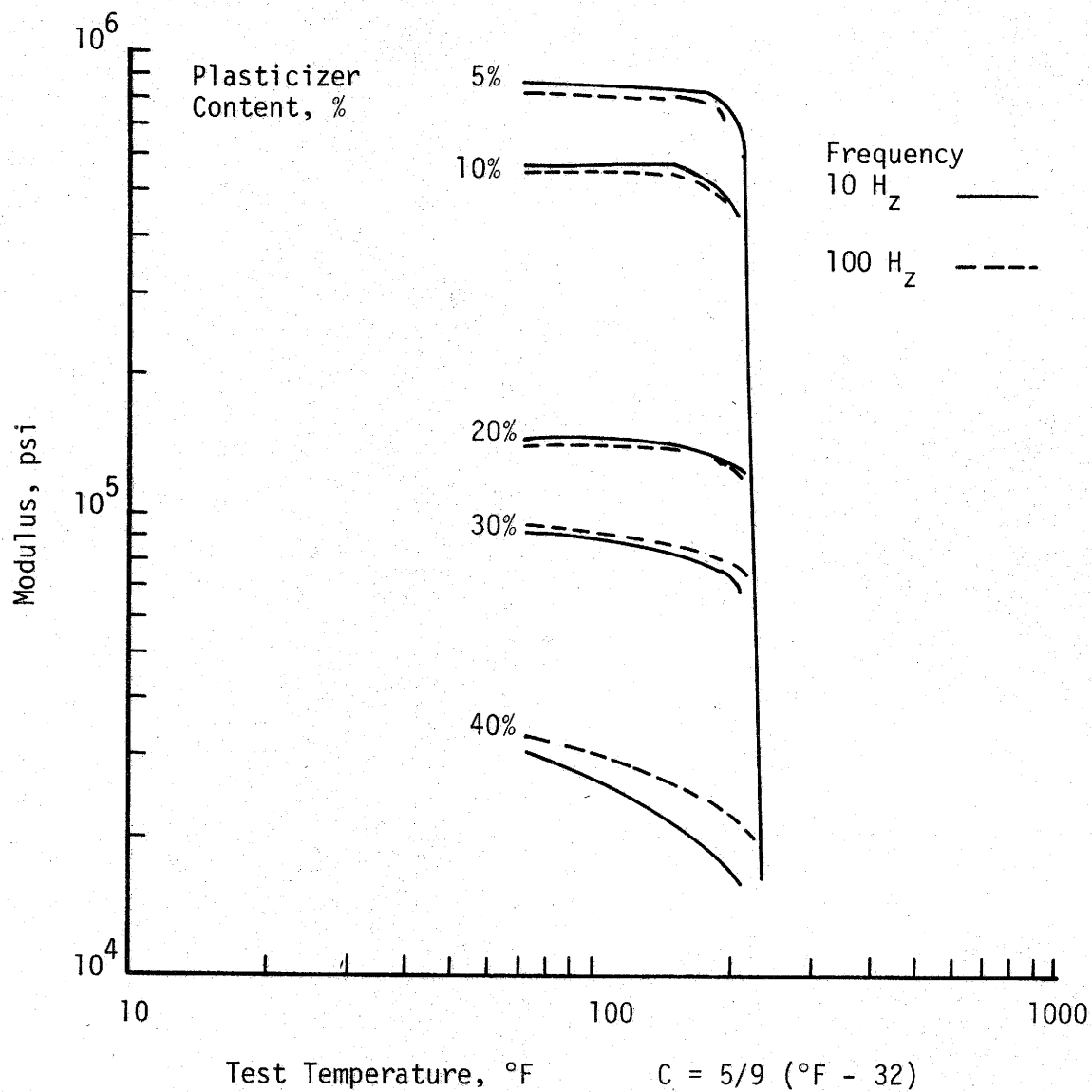


Figure 22. Effects of Duomorph Excitation Frequency on Modulus versus Temperature and P/S Ratio of Plasticized Sulfur Binders.

results appear to be consistent with the elastic behavior encountered in the creep tests as reflected in the near zero shift established for the temperature ranges employed. One possible explanation can be attributed to the length of time which had elapsed between the resilient modulus tests and those run on the Duomorph which could have permitted the binder to age harden. Because of the sample preparation problems discussed earlier, the time lapse between the two tests was about seven months. If this were the case the Duomorph's ability to indicate changes with age would have at least been partially demonstrated.

Tests were also run in which hot liquid binder at 265°F (130°C) was permitted to cool down to 200°F (93°C) at a rate of about 5°F (2.8°C) per hour. This was done to see if the Duomorph could detect any phase changes as the binder went from liquid to solid. There was no indication in the materials response which could be related to the transition from the monoclinic to the orthorhombic allotrope of sulfur. Throughout the transition range, the change in stiffness was significant, as would be expected, but uniform and continuous.

4.0 CONCLUSIONS

A number of modified sulfur binders with varying amounts of plasticizer (5, 10, 20, 30 and 40 percent, respectively) and sulfur concrete test specimens prepared with these binders were furnished by the Bureau of Mines Boulder City Engineering Laboratory and investigated to determine their potential as highway paving

It was found that for sulfur concrete specimens with thicknesses exceeding 3 in. (7.6 cm), sample preparation must be accomplished in more than one layer. This was necessary for compression test specimens which are usually cast into 4 in. (10.2 cm) diameter and 8 in. (20.4 cm) high or 6 in. (15.2 cm) diameter and 12 in. (30.4 cm) high cylinders. The California DOT Test Method No. 304 which is routinely used by TTI for preparation of field control samples for stability, cohesivity and moisture susceptibility tests was found to be acceptable for making larger size samples. This procedure calls for both rodding and tamping to offset any weak zones at the interface between layers. For smaller sample sizes, standard Marshall or Hveem, procedures for bituminous mixtures can be used. Specific gravity, compression strength and air voids of mixtures plasticized at 5 percent were consistent with those reported by the Bureau in their studies.

Characterizations of the behavior of sulfur concretes plasticized at 5, 10, 20, 30 and 40 percent were analyzed using VESYS IIM. Performance characteristics such as rutting potential, slope variance, resistance to fatigue cracking and serviceability index were shown to be equal or superior to conventional paving systems.

Freeze-thaw tests were also run which resulted in a failure between 35 and 69 cycles. Poor surface characteristics of the samples could have contributed to a more rapid decrease in durability. Additional testing is recommended to better define the durability of these systems.

Compressive strength values were indirectly related to the amount of plasticizer in the binder. Concretes plasticized at 5 and 10 percent were comparable with high grade portland cement concretes. The 40 percent system had compressive strengths more in agreement with asphaltic concretes.

A series of stringent characterization tests were run to provide input to a state-of-the-art linear viscoelastic pavement systems analysis (VESYS IIM). In terms of serviceability, roughness characteristics, fatigue cracking and rutting potential, the sulfur concretes should perform very well in a highway pavement system.

The Duomorph was demonstrated to have the capability of monitoring rate and aging effects in plasticized sulfur binders. A test procedure was developed along with auxiliary equipment to determine changes in material stiffness with loading-rate and temperature. The potential for monitoring behavioral changes with storage time was also demonstrated.

5.0 RECOMMENDATIONS FOR FUTURE WORK

As a result of the investigation just completed the following additional work is suggested.

1. Investigate the fatigue response of sulfur concrete in the critical controlled-strain mode, and through simulation testing evaluate the crack propagation characteristics (fracture) of the various mixtures.
2. An optimum mix design rational needs to be developed which improves on the current Marshall or Hveem methods used for asphaltic concretes. The recommended effort would optimize mixtures on the basis of their combined resistance to thermal cracking, rutting, and fatigue rather than the simplified indexing of performance by stability numbers determined under a single temperature environment.
3. The aging characteristics of the binders during storage need to be quantified and, if necessary, steps to minimize these effects need to be developed.
4. Investigate other fillers such as fibers and aggregate coatings to enhance freeze-thaw durability.
5. Aggregate-bonding interaction and its ultimate effect on performance under shear-induced loads need to be studied further.

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APPENDIX A

Paper - Reference 9

"The Duomorph - An In-Situ Viscoelastic
Characterization Transducer" by Saylak, D.,
Noel, J.S. and Boggess R.

The Duomorph – An In-Situ Viscoelastic Characterization Transducer

D. Saylak, J. S. Noel and R. Boggess, USA

ABSTRACT

This paper describes a device which can be used to measure rate-dependent properties of materials in both laboratory and field environments. The Duomorph has been designed for use either on the surface or embedded in materials with moduli ranging from 10 to 10⁶ psi. Construction and operating details are presented along with graphical solutions to permit rapid reduction of field data into elastic and viscous components of the complex modulus. The potential for extending the use of the Duomorph to monitoring cure cycles, and aging of filled and unfilled polymeric systems is also discussed.

ZUSAMMENFASSUNG

Dieser Artikel beschreibt eine Anlage die gebraucht kan werden zum messen Gang-Abhangiger Eigenschaften von Material im Laborator sowie in praktische Umweltbedingungen. Der Duomorph ist entworfen geworden zum gebrauch entweder auf der Oberflache oder innerhalb Material, mit Modulus Reichen von 10 bis zu 10⁶ psi. Konstruktion und Betriebsdaten werden vorgestellt zusammen mit Graphische Loesungen die schnelle reduktion erlauben von praktische Messwerte zu Elastische und Viskoese Daempfung Komponenten des Komplex Modulus. Das Potential zur Erweiterung der Anwendung des Duomorph zum Registrieren des Haerten und Vergueten von auffgefuelle und nicht auffgefuelle Polymern wird im Betrag genommen.

ABREGE

Cet article decrit un mecanisme qui peut etre utilise pour mesurer le taux des proprietes dependentes des materiaux dans un milieu de laboratoire aussi bien dans des milieux environnants. Le Duomorph a ete concu pour usage ou bien dans la surface ou a l'interieur des materiaux avec des modules rangeant de 10 a 10⁶ psi. Les details de structure et de fonctionnement sont presentes avec des solutions diagrammees a fin de permettre la reduction rapide de donnees pratiques dans des composants d'elasticite et de viscosite du module complexe. Le potentiel pour itendre l'usage du Duomorph comme moniteurs des cycles de cure et d'affaiblissement progressif de systems polymeriques remplis et vides, est egalement decrit.

1. Introduction

Often it is necessary to monitor physical or structural changes which are occurring in a material after it has been in service. Sometimes this can be accomplished by taking samples during fabrication and storing them in simulated environments for periodic testing at some later date. This form of surveillance testing gives rise to the question "is the separately stored sample representative of the material in the field?". If there is a gross difference anticipated, field samplings must be taken.

In the case of polymeric systems, changes in a material's structural integrity are usually associated with changes in stiffness since this parameter provides an index of its current deformation and load carrying capability. This is especially true in evaluating performance ratings for asphaltic concrete highways although similar treatments have been employed for solid propellants [2], fine-grained soils, rubbers and plastics.

Two approaches most commonly used to generate stiffness data on asphalt pavements are by coring and deflection measurements. The former procedure entails the drilling of a cylindrical core which can be taken back to the laboratory for testing. The laboratory procedure for determining the dynamic stiffness, also called the Resilient Modulus M_R , was developed by Schmidt [3]. This test is carried out by means of measuring the resultant strain produced by a high rate pressure pulse delivered diametrically to a cylindrical specimen. In the former, deflections are measured by means of truck mounted devices which apply a programmed load through a ram pushing downward on the surface. Either geophones or LVDT's [4] are used to monitor the shape of the resulting deflection basin.

The single valued stiffnesses generated by the above methods are sufficient for use in elastic layered pavement analysis computer codes [5] but were found to be incapable of satisfying the input requirements of recently developed viscoelastic programs [6].

Both of the approaches mentioned above are currently being altered to provide a more complete characterization of the rate-dependent properties of the pavement material. The device to be described presents a technique to generate the viscoelastic characterization required by the new computer codes. It should be noted that although the data presented was taken on asphaltic concrete samples, the techniques and data reduction methods discussed below can be extended to other polymeric materials.

The Duomorph – An In-Situ Viscoelastic Characterization Transducer

D. Saylak, J. S. Noel and R. Boggess, USA

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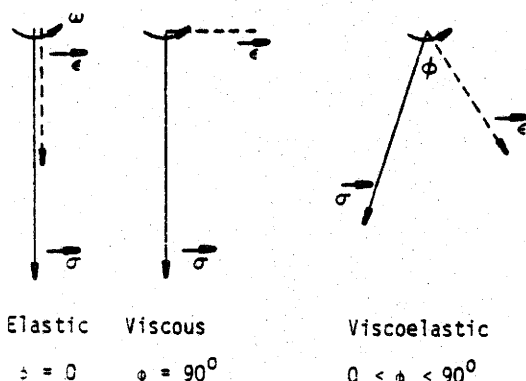
The single valued stiffnesses generated by the above methods are sufficient for use in elastic layered pavement analysis computer codes [5] but were found to be incapable of satisfying the input requirements of recently developed viscoelastic programs [6].

Both of the approaches mentioned above are currently being altered to provide a more complete characterization of the rate-dependent properties of the pavement material. The device to be described presents a technique to generate the viscoelastic characterization required by the new computer codes. It should be noted that although the data presented was taken on asphaltic concrete samples, the techniques and data reduction methods discussed below can be extended to other polymeric materials.

2. Viscoelastic Response

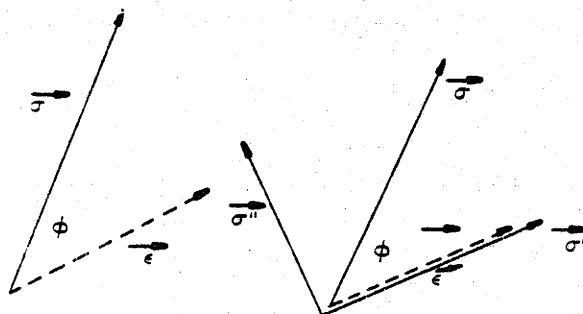
The viscoelastic properties which can be determined by the Duomorph include; complex modulus (E^*), its associated elastic (E') and viscous (E'') components and loss tangent ($\tan \phi$). These two parameters are determined under dynamic conditions (i.e. where the stress or strain loadings are oscillatory functions). The resulting parameters measured using such perturbations are functions of frequency rather than time.

If a material is subjected to a sinusoidal stress loading function its strain response will reflect the nature of its mechanical properties. If the stress and resulting strain are in phase with each other (i.e. phase angle $\phi = 0$; independent of frequency and elapsed time) the material's behavior is categorized as elastic. If on the other hand the strain is 90° out of phase with stress, the material is considered to be viscous. For phase angles in the range of $0^\circ < \phi < 90^\circ$ the material is characterized as viscoelastic, the degree of which is assessed by the magnitude of ϕ . These three classifications are depicted below.



Now consider a sample of viscoelastic material subjected to a sinusoidal stress. The stress and its strain response at any time can be represented using the technique of rotating vectors. In the figure given below, the magnitude of the stress vector $\vec{\sigma}$ represents the maximum of the stress applied to the sample which is being loaded at a frequency, ω . It is obvious from the figure that the two vectors do not coincide. The strain lags the stress by the angle ϕ which is also called the loss or lag angle.

The absolute modulus of the material $|E|$ is defined as the magnitude of the stress vector divided by the magnitude of the strain vector. Quite often it is convenient to separate the viscoelastic response into "in-phase" (elastic) and "out-of-phase" (viscous) components. This is done as shown below.



Here the projection of $\vec{\sigma}$ onto $\vec{\epsilon}$ yields $\vec{\sigma}'$ the component of $\vec{\sigma}$ in phase with the strain while the projection of $\vec{\sigma}$ on the axis perpendicular to $\vec{\epsilon}$ yields $\vec{\sigma}''$, the component out of phase with strain. The elastic and viscous moduli, E' and E'' respectively, and the loss tangent, $\tan \phi$, can be computed as follows:

$$E' = \frac{\sigma'}{\epsilon} \quad E'' = \frac{\sigma''}{\epsilon} \quad (1)$$

$$\tan \phi = \frac{\sigma''}{\sigma'} = \frac{E''}{E'} \quad (2)$$

$$E^* = E' + iE'' \quad (3)$$

In the last expression, the viscoelastic modulus has been shown in its complex form where the out of phase component, E'' , is made the imaginary part and E' the real part of E^* .

The values of E' have a direct relation to the degree of cross linking in a polymer which in turn gives the material its elastic characteristics. If the material ages or experiences changes in its cross link density this will result in a change in the magnitude of E' . The values of E'' reflect any changes in polymer chain structure brought about by chain scission, hydrolysis, plasticizer migration, etc. Since both types of activity can be occurring simultaneously during the life of the material the change in magnitude of the loss tangent with time can be an indication of the primary mechanisms involved. Hence changes in these properties represent convenient parameters for monitoring aging in polymeric systems.

3. The Duomorph - Design and Operation

The Duomorph sensor consists of two radially expanding piezoelectric (PZT) ceramic crystals bonded together into a circular bending plate. When excited by an electric field the plate is distorted into a parabolic surface due to the radial expansion and/or contraction of the crystals. As the polarity of the excitation is cycled the disc goes into reversed bendings. Figure 1 shows the general arrangement of the sensor.

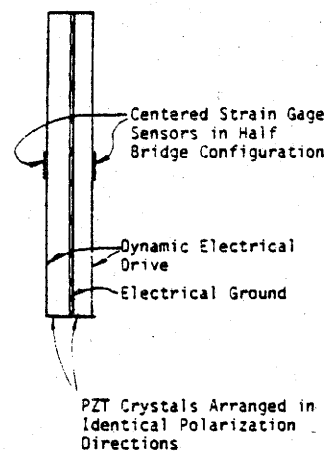


Fig. 1. Duomorph Sensor

The two PZT elements are bonded together using an epoxy adhesive. Strain gages cemented to each face of the sensor form a bridge which can measure very small bending strains. If the device is to be used on hard surfaces or in hostile environments a thin layer of epoxy should be applied to the sensor surface to protect the delicate strain gages.

The sensor must be tightly coupled to the specimen so that all forces and motion are transferred into the material being tested. This can be done either by totally embedding the sensor into a material or by forcing it onto a surface with sufficient pressure to insure good contact. For the surface-mounted sensor to function properly the surface must be smooth since surface irregularities or debris will adversely affect the performance of the Duomorph.

The Duomorph sensor is sensitive to a modulus range of 1 decade; e.g. 700 MPa to 7000 MPa (10^5 psi to 10^6 psi). This means that a Duomorph must be sized for a particular range of modulus. For wide ranges of E more than one sensor may be required. Since the mechanical properties of the ceramic material remain nearly constant, designing a Duomorph of the proper stiffness is only a matter of selecting the proper thickness-to-diameter ratio.

The transducer is made by embedding the Duomorph sensor in the surface of a mass of low modulus (≈ 300 psi) silicone rubber which is contained in an aluminum cylinder. The diameter of the cylinder should be large enough to minimize end effects. The silicone rubber distributes the pressure uniformly over the surface of the sensor and holds it firmly against the material to be tested. A schematic drawing showing the components which make up the transducer is shown in Figure 2.

The Duomorph sensor used in the work discussed in this paper was designed for use on asphaltic concrete. These materials can have moduli ranging from 700 to 7000 MPa (10^5 to 10^6 psi) depending on the type of ingredients used in the concrete, its temperature or rate of loading.

It was found that in order to get deflections in asphalt large enough for satisfactory measurement, a driving voltage of ± 250 V is desirable. Because of the capacitive nature of the Duomorph sensor, the power required for driving increases with frequency. A schematic of the Duomorph circuitry is shown in Figure 3.

Early in the developmental program it was found that a very large error was introduced into the strain gage signal by capacitive coupling of the excitation voltage. Due to the bridge arrangement and the fact that the two outer surfaces were oppositely charged, an error signal was coupled into the strain gage bridge. This error signal was in phase with the expected output signal. Two corrective actions were taken to eliminate this annoyance. A symmetrical configuration was adopted. This configuration allowed the outer surfaces to be kept always at the same potential, thus eliminating any capacitively-coupled signal. The other corrective measure was the use of AC excitation to the bridge.

The amplifier used has a band pass of 2500 Hz and is capable of resolving strain of less than $1.5 \mu\text{-in./in.}$, with typical signal strain levels of $200 \mu\text{-in./in.}$

4. Data Reduction

The method for reducing the Duomorph output to find the dynamic properties of viscoelastic materials relies on the results of an analysis published by Schapery in 1976 (see Reference 7). Typically the driving voltage and the output of the strain gages are both fed into an oscilloscope which displays them on the cathode ray tube as the ordinate and abscissa, respectively. An example of such a plot

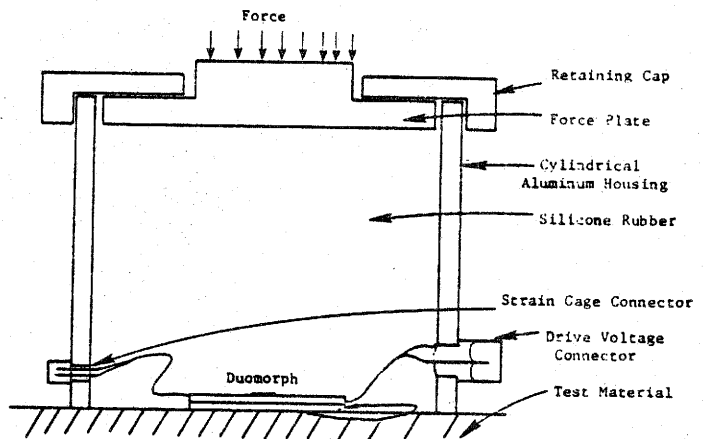


Fig. 2. Assembly for surface measurements

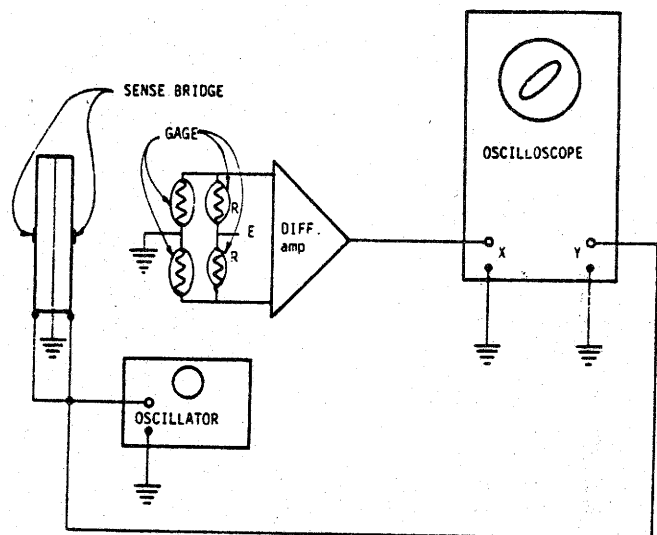


Fig. 3. Schematic diagram of Duomorph test apparatus

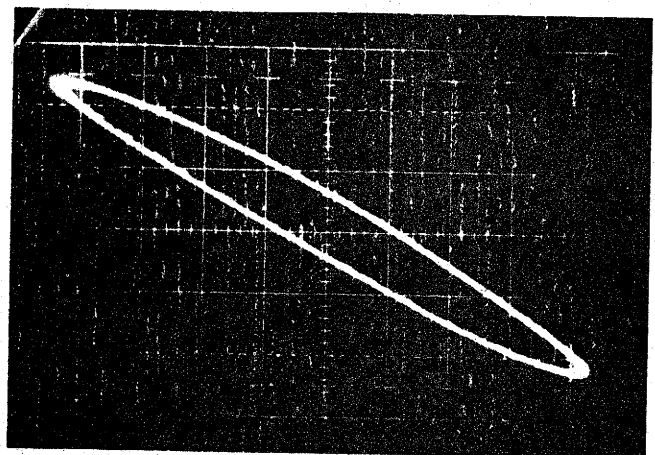


Fig. 4. A typical Lissajous plot

conventionally referred to as a Lissajous curve, shown in Figure 4. The elliptical shape is characteristic for viscoelastic materials wherein the slope and area are related to stiffness and the inherent damping characteristics of the material.

If the strain is exactly in phase with the voltage, as would be the case for elastic behavior, the ellipse would degenerate into a single sloping

Figure 7 shows the real and imaginary moduli of an asphaltic concrete pavement material cored from Texas Farm-to-Market Road 493. All tests were performed in the laboratory at 70°F and reflect the type of behavior that can be expected of asphalt. The real part of the modulus, E' , increases linearly on the log-log plot versus frequency. The imaginary part of the modulus, E'' , also increased over the range of frequencies tested. The rate dependence shown indicates the material's behavior over this frequency range is viscoelastic.

The Resilient Modulus, M_R , data generated at different temperatures are also shown on the graphs. These points are plotted at 2Hz which corresponds to the width of the pressure pulse used in the resilient modulus test [3]. Since M_R is an elastic modulus its value should be expected to correspond to E' at the same temperature and loading rate. The good agreement between M_R and the E' at 70°F and 2Hz is readily apparent.

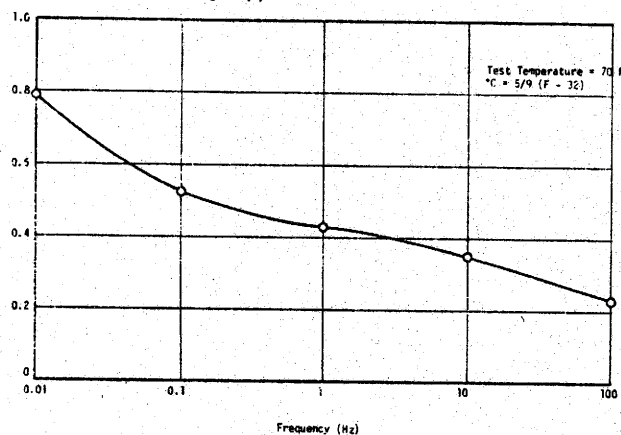


Fig. 8. Loss tangent vs. frequency data from asphaltic concrete samples of Texas Farm Road 493

Figure 8 shows the relationship between loss tangent and frequency. The reduction in $\tan \phi$ with frequency is characteristic of viscoelastic behavior. As the frequency continues to increase the viscous component will vanish and $\tan \phi$ will approach zero.

5. Conclusions

This work has demonstrated that the Duomorph may be used as a sensitive device for obtaining the dynamic moduli and loss tangents of asphalt pavements. The test equipment is compact, portable and convenient to set up and use in the field. The necessary data can be collected rapidly and non-destructively so that the extraneous effects of handling and sample preparation are minimal.

The results cover a range of practical frequencies (loading rates) characteristic of those induced by automobile and truck traffic.

The graphical solutions provide a means of rapidly reducing field data to the real and imaginary modulus components. Such rapid reduction capability makes it possible to compare data in the field and make on-the-spot test checks if necessary.

The numerical values of the moduli measured with the Duomorph compared favorably with those measured using the Resilient Modulus Test, the routine test for dynamic modulus used by highway engineers. This adds to the confidence that can be placed in results and provides encouragement for further applications using the device. Work is now in progress to extend the use of the Duomorph to other materials such as fiber reinforced composites. In this sense the device will be used to monitor structural changes during cure from which the basis for optimizing process variables can be established.

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line. On the other hand, if the strain is 90° out of phase with the voltage, the area enclosed by the ellipse becomes a maximum.

From a material standpoint it is necessary to interpret the ordinate and abscissa in units that can be related to mechanical properties of the material being tested. The abscissa can be simply calibrated into strain units by using the conventional application of a known shunt resistance to the strain gage bridge in conjunction with the gage factor. Thus the abscissa can be directly interpreted as the strain on the surface of the disc.

The interpretation of the driving voltage is more difficult, and is circumvented by using a ratio of driving voltage in air to that against the material. When viewed in this manner the driving voltage can be considered to be linearly related to the equivalent line moment which if uniformly distributed around the periphery of the sensor would force the same shape changes. In practice one should use the same driving voltage for establishing the shape of the ellipses in air (one for each frequency) as when in contact with, V_0 the material being tested, V_C .

If the driving voltages are the same then the ratio of the total strain excursion with the sensor in contact with the specimen, ϵ_0 , to the total strain excursion in air, ϵ_c , yields to a number reflecting the degree of restraint caused by the sample material. If the driving voltages are different, the ratio must be modified by the ratio of the driving voltage, thusly

$$\frac{M_C}{M_0} = \frac{\epsilon_c}{\epsilon_0} \frac{V_0}{V_C} \quad (4)$$

The numerical value of this ratio is entered as the ordinate of Figure 5 which when transposed to the abscissa yields M' a parameter which relates the gage and material stiffnesses.

Knowing M' it is next necessary to deduce the loss tangent, $\tan \phi$. This can best be done using the relationship

$$-\sin \theta = \frac{1}{2} \left(\frac{V_1}{V_2} + \frac{H_1}{H_2} \right) \quad (5)$$

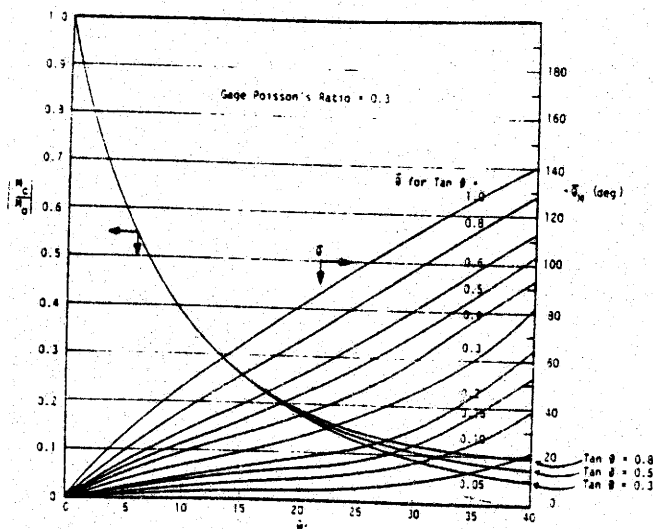
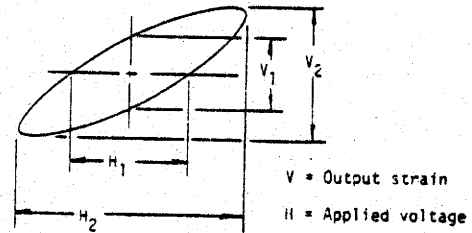


Fig. 5. Curves for reducing the output of the Duomorph to the real and loss tangent components of the dynamic modulus



$$-\sin \theta = \frac{1}{2} \left(\frac{V_1}{V_2} + \frac{H_1}{H_2} \right) \quad \frac{M_C}{M_0} = \frac{V_2 \text{ (material)}}{V_2 \text{ (air)}}$$

Fig. 6. Graphical determination of bending moment ratio and θ_m from Duomorph output

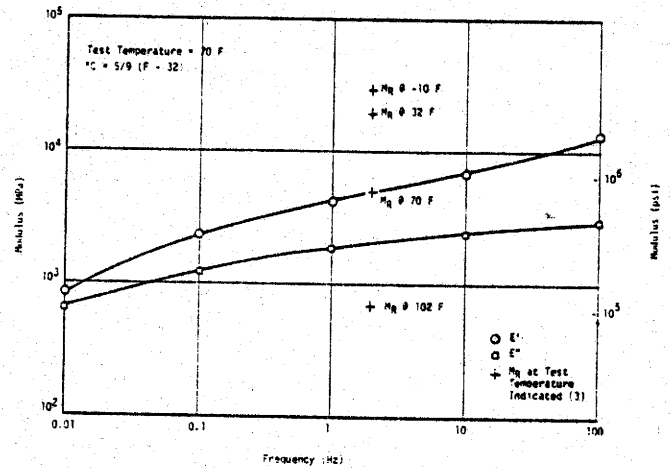


Fig. 7. Comparison of complex modulus and resilient modulus data from asphaltic concrete samples of Texas Farm Road 493

where the values of V_1 , V_2 , H_1 and H_2 are measured from the Lissajous curve as shown in Figure 6. If θ is determined at each frequency, both in air, θ_0 , and when pressed against the material being tested, θ_c then θ_m is determined by

$$-\theta_m = -(\theta_0 - \theta_c) \quad (6)$$

This value is used to enter the right hand scale of Figure 5.

Once the point defined by the coordinates (M' , θ_m) is found the appropriate family of curves yields $\tan \phi$.

The real part of the modulus of the asphalt can be computed using the equation

$$E' = \frac{8}{3} (1-\nu^2) M' \frac{E_s h_s^3}{12 a^3 (1-\nu_s^2)} \quad (7)$$

where the subscript s indicates that the thickness, h , the radius, a , and Poisson's ratio ν apply to the sensor. And finally E'' is computed using

$$E'' = E' \tan \phi \quad (8)$$

For viscoelastic materials the complex modulus is a function of the frequency. So both E' and E'' must be determined for discrete frequencies and plotted as shown in Figure 7. These data were generated on samples taken from two asphaltic pavements in Texas.

