A New Approach in Asphalt Rheology

HERBERT E. SCHWEYER and J. CARLOS BUSOT, Department of Chemical Engineering, University of Florida

This paper presents arguments for the need of using rheology in specifications for paving asphalt. In particular, the paper discusses the need for determining the deformation characteristics employing asphalt invariant parameters with the objective that such results will be useful in bridging the gap from the scientific measurement of asphalt to the utilization in the design of better flexible pavements.

A review of recent publications pertinent to the subject is included and refers in particular to the more sophisticated considerations of rheology phenomenological behavior. From this background, a new method of characterizing the rheology of asphalt is proposed as being more rigorous and, it is hoped, more useful for design purposes.

The procedure described uses the Instron testing machine with an environmental chamber and a compression test in which the basic concept of compressibility is utilized for evaluating three rheological parameters. These parameters are the stress-strain relation at a zero rate of strain condition, a measurement of consistency related to classical viscosity, and a characteristic time, which is a measure of the memory of previous strains that the material retains and that influence its rheological behavior. These three independent material parameters are sufficient to describe the asphalt for all rheological purposes. Line diagrams are presented to illustrate the procedure and an example of the proposed analysis for an 85 to 100 penetration asphalt is given.

•AS AN introduction, we wish to emphasize that this paper presents mainly new concepts for asphalt rheology with only limited data. Its purpose is to reiterate, for the record, the need for new approaches and to promote discussions and suggestions in using compression techniques.

NEED FOR ASPHALT RHEOLOGY

The flow properties of asphalt have been measured in a variety of ways from the time it was first used as an engineering material. Originally these methods of measuring rheology were empirical in nature; examples are the penetration test, ductility test and softening point, which subsequently were studied from theoretical aspects and were found to have serious limitations. It is necessary to study the flow properties of asphalts in order to have some measure of consistency and uniformity of materials being used in a given application.

Consideration of this has led to emphasis on studying flow properties using invariant parameters. Although much progress has been made, methods used today are still inadequate for the intended purposes. In fact, it may be unequivocally stated that no specification test for asphalt, as measured today, can be employed directly for the

Paper sponsored by Committee on Characteristics of Bituminous Materials and presented at the 48th Annual Meeting.

design of bituminous pavements. Thus, there is a great need for a suitable test that can be run by technicians and that can be interpreted correctly in terms of true rheological phenomena and be of immediate application to design.

The review of literature will primarily refer to certain early articles and also to more recent publications that contain bibliographies from which one may obtain a more complete history. The basic concept in this paper relates to thermodynamic considerations that Mack (1, 2, 3) was one of the first to recognize. Other investigators have studied, with varying degrees of success, the application of physicochemical principles to asphalts. Perhaps one of the most important developments is the application of the superposition principle to study the rheological behavior of asphalt over a wide temperature range. Shoor, Majidzadeh and Schweyer (4) present an illustration of this along with certain other applications of newer techniques. Their work is by no means novel, but it does include a number of different types of asphalts for study and the references may be consulted for other investigations in the field. Certain specific references selected by us as being quite pertinent to the studies of the nature of asphalt deformation over a wide range of temperature include Heukelom (5), who discussed the experimental aspects of certain deformation tests; Schmidt and Santucci (6); Sisko (7); Majidzadeh and Schweyer (8); and Moavenzadeh (9). These men as well as others have worked with asphalts in those temperatures and conditions which produce asphalt fracture. Krchma (10) proposes a new type of softening point as a measure of the rheological characteristics of asphalts in the intermediate temperature range, where the temperature at which the asphalt demonstrates certain rheological properties is considered a critical characteristic.

These new approaches have been presented during approximately the last ten years and include the works of Gaskins, et al (11) and Brodnyan (12), who have provided certain methods to approach the asphalt rheology problem. The work here will be based on this background. The basic rheology concepts and rational mechanics approach used can be found in Slattery (13), Coleman, Markovitz, and Noll (14), Middleman (15), Truesdale (16, 17), and Lodge (18).

CONCEPT OF APPLIED RHEOLOGY AND SPECIFICATIONS

The ultimate goal of rheological studies on paving asphalts is to apply the results to specifications. There are really two types of specification requirements for these materials, and they must be kept in proper perspective. First, it is necessary to know the requirements for asphalt material to behave properly under selected process conditions. This is a process specification. An example is the consideration of deformations in pavements under compaction as influenced by the viscosity of the asphalt. Second, there are material responses in the form of rheological behavior that are a function of the composition of the asphalt. This is a product specification.

In general the procedures for testing flow characteristics of asphalts above 122 F present no problem because paving asphalt cements of 85 plus penetration are essentially Newtonian. These results provide numerical values for viscosity (assuming no complications due to hardness) that can be compared with required specifications for the process conditions—mixing and compaction requirements. However, as lower temperatures are required for consideration in either the process or product specification categories, it becomes necessary to consider more complicated methods of measurement to obtain significant numerical values. These test temperatures may range down to 0 F and, accordingly, special equipment is required. As noted, work along these lines has been done and the resulting development of highly sophisticated equipment permits considerably more application of fundamental rheological principles to analysis of the data. Furthermore, the ultimate use of such data for translation into highway pavement design is a very necessary goal for improved understanding of the rheological behavior of flexible pavements.

Majidzadeh (19) has indicated a direction in which the necessary data on rheology of the asphalt component may be used for rational design of highway pavements in connection with sand-asphalt mixtures where three design criteria are noted—stability, durability, and resistance to fatigue. However, as far as is known to us, the rheological values have never been translated into practical applications for the design of bituminous

Figure 1. Instron floor model.

paving mixtures. It would appear that the reason for this lack of use of such information is that to date all rheological data are not presented as invariant parameters. Such data cannot be used because the data presented have as yet not considered the basic constitutive assumptions defining the phenomenological behavior of the asphalts. An understanding of these relationships will provide the bridge between specifications and the rational design of bituminous pavements.

A SUGGESTED NEW APPROACH IN ASPHALT RHEOLOGY

Researchers at the Asphalt Laboratory at the University of Florida have, for a number of years, studied the rheological behavior of bituminous materials. The initial investigations began with the sliding plate viscometer developed by Shell Oil Company. This was followed by the cone and plate viscometer developed by Sisko of the American Oil Company. Concurrently, work was carried out using low-temperature creep tests on asphalt, and initial studies were started using an Instron rheometer. Because of the complicated nature of the viscoelastic flow of asphalt cements, it soon became apparent that all of these instruments had certain limita-

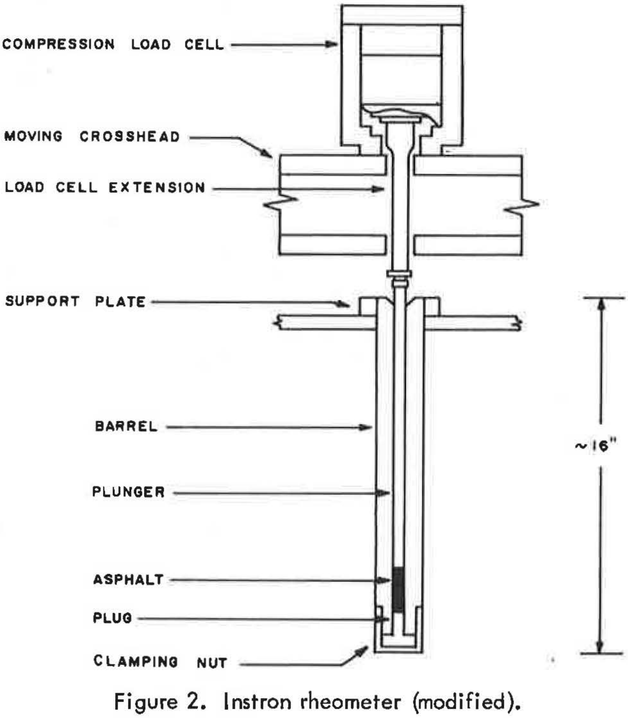

Figure 2. Instron rheometer (modified).

Figure 3. Rheometer assembly.

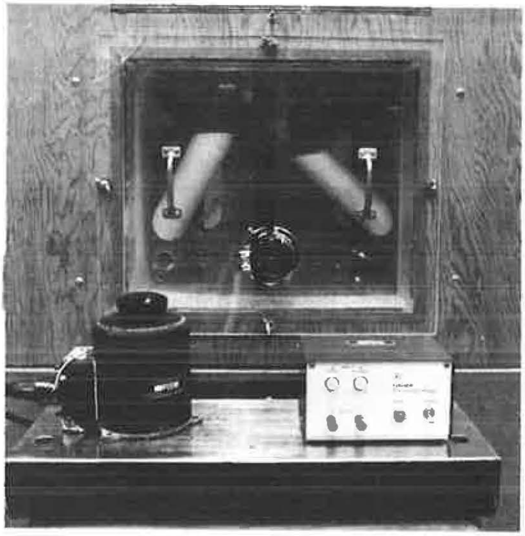

Figure 4. Environmental chamber.

tions. As a result, attention was focused on using the Instron testing equipment in order to measure a time constant characteristic of the flow of asphalt at temperatures of 32 F (0 C). Study of the stress relaxation indicated a possible approach for measurement of this characteristic time in addition to evaluating a viscosity parameter. For this purpose an Instron machine was used (Fig. 1). An Instron rheometer with a plug replacing the orifice at the bottom (Fig. 2) was utilized to study stress relaxation. The components of the barrel assembly are shown in Figure 3, and the environmental chamber for the testing machine is shown in Figure 4.

COMPRESSION OF ASPHALT

The compression of asphalt in a confined space as described here is new, as far as known. Certain work is being carried out in the University of Florida laboratory to evaluate the coefficient of compressibility at different temperatures. However, there are certain considerations in such experimental work that must be recognized. For example, the compression load under strain may be studied with the drag on the wall being included. This requires evaluation after an equilibrium stress value is attained at a given strain, which extends the time of an experiment. Conversely, the walls of the confining barrel may be lubricated, permitting study of "internal" viscosity and faster determination of equilibrium data. Each method has value in providing information concerning the rheology of asphalt, as will be demonstrated with examples and preliminary consideration of the significance of the results.

Deformation With Drag

If an asphalt is placed in a confined geometry and subjected to deformation, a curve such as shown in Figure 5 is obtained. If the deformation is stopped, a stress relaxation will occur, declining to some equilibrium stress value denoted by τ_0. (This may be considered the value obtained at a zero deformation rate.) However, the machine also deforms as shown in Figure 5 and, being essentially elastic, shows a time deformation curve with only a very small relaxation of stress. The infinite time asymptote designating τ_0 assumes there is no residual stress τ_r above τ_0. For the real case there may be a definite residual stress τ_r, which may or may not be significantly higher than the equilibrium value.

The relations for the deformations are shown in an exaggerated scale in Figure 6, where the difference between the total deformation ΔR and the machine deformation ΔM represents the deformation in the sample, ΔS. In a cylindrical section it is assumed that radial changes in the machine barrel are not significant. An illustration of the time-deformation curves is shown in Figure 7, which also shows the relationships involved.

In the actual performance of a test the samples are preheated in a tared container and poured into the bottom of the heated barrel to a predetermined length of 1 to 2 inches. The plug is inserted and capped and the barrel assembly placed in the environ-

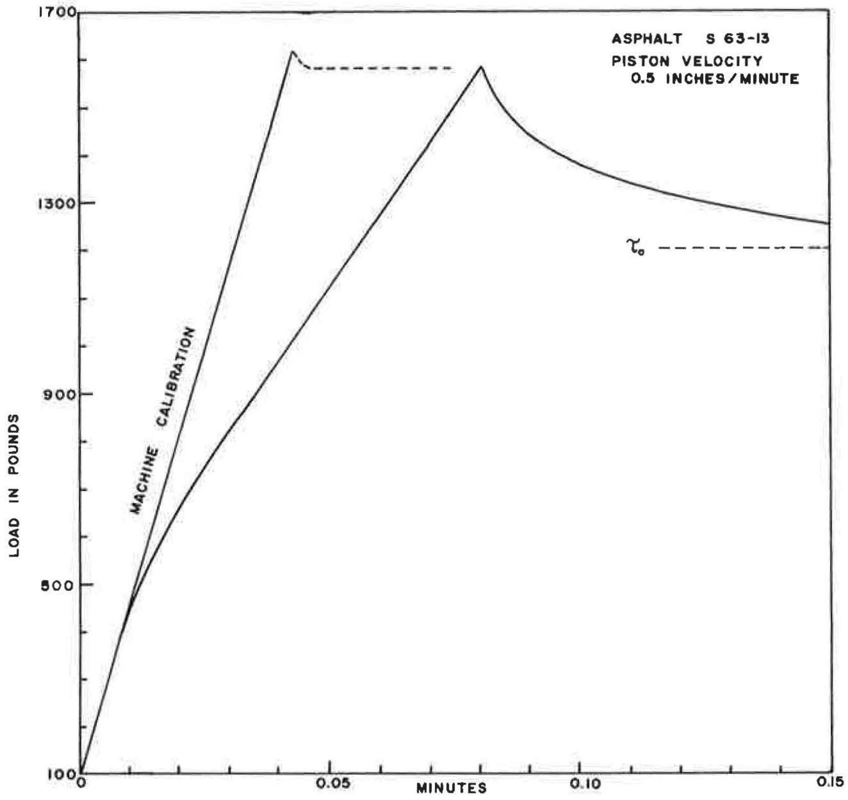

Figure 5. Deformation curve with drag.

mental chamber. The exact amount of asphalt is determined from the residual weight of the tared container and contents.

After the assembly has been cooled to the test temperature, an initial small set stress is applied to remove backlash in the assembly. Application of stress after the temperature has been lowered below 10 C virtually eliminates leakage of the sealing O-rings. Checks have been carried out by determining the volume of asphalt before and after conducting the experiments. Initial and final volumes check within ±0.03 percent, which is approximately 1 percent of the measured deformation during experiments. Starting from this point the machine crosshead is run at different deformation rates and times, and the stress is recorded. The sample is then held and stress relaxation permitted to approach an equilibrium value. Different crosshead velocities are used on the same sample by permitting the sample to rebound and by repeating the deforming procedure. It should be obvious that a given amount of strain will require less time at a higher crosshead speed, but these can be normalized for a given

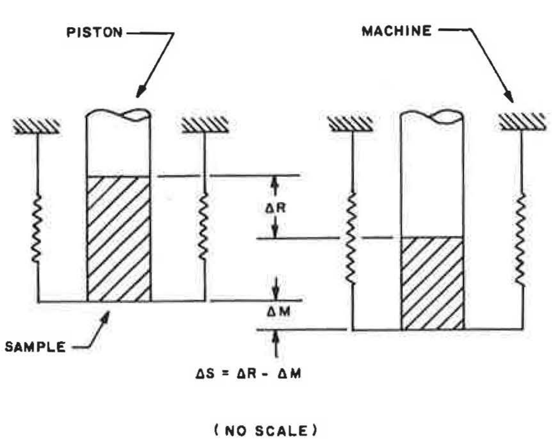

Figure 6. Model for compression (no scale).

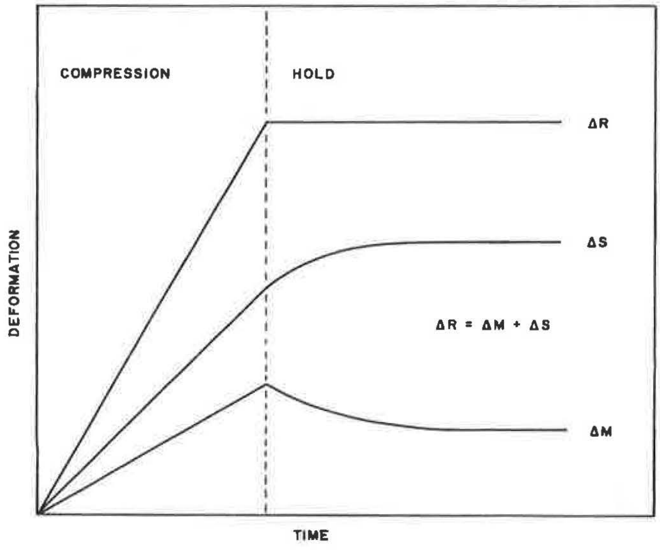

Figure 7. Comparison of strains.

amount of strain as shown in Figure 8. These are corrected strains obtained from the relationships of Figure 5. The machine calibration curves (stress vs strain is the same regardless of the crosshead velocity) are superimposed on the actual experimental recordings. At a given stress the strain is determined as $\Delta S = \Delta R - \Delta M$, the data plotted in Figure 8. The different strains are normalized from the time-deformation curves recorded by the machine.

The zero-rate curve shown in Figure 8 is for the equilibrium values discussed and represents the thermodynamic reversible change in density with pressure at constant temperature 32 F. The other curves represent the real situation, and since the deformation coordinate is related to density, the slopes represent the dynamic expressions for change of stress with respect to density of $(\partial\tau/\partial\rho)_T$, which is related to internal cohesive forces.

As noted in Figure 8 on asphalt S63-13, during deformation at 32 F there is a drag effect where the asphalt is in contact with the walls. Thus, excess stresses above the zero rate may be utilized to evaluate this viscous drag effect. Theoretically, the zero-rate (or reversible) curve is a measure of the elasticity of the material at a given temperature. It can be determined from procedures shown in Figure 5.

The excess stress for different rates of deformation is not constant (parallel to the zero-rate line). This could be due to a pressure-dependence of the material parameters as well as other possible nonlinear phenomena. From other work carried out in this laboratory there is evidence that the measured consistency (viscosity) may be influenced by changes in density. Figure 8 shows that pressures up to 15,000 psi are approached and these could definitely affect the rheology of the material.

In addition, part of the deviation of the curves from the zero rate in Figure 8 may be caused by memory effects. The history of deformations may determine the state of stress at the time of consideration. The memory (influence of past deformations) of real materials is a fading memory. This means that a deformation that occurred a long time in the past will have little influence on the present deformation regardless of the magnitude of the earlier deformation. The converse is true for materials that have a permanent memory.

A perfectly elastic material will permanently remember only its unstressed state (τ is unique function of strain from a reference state). Conversely, a Stokesian fluid will have such a short memory that only the deformation instantly before the present

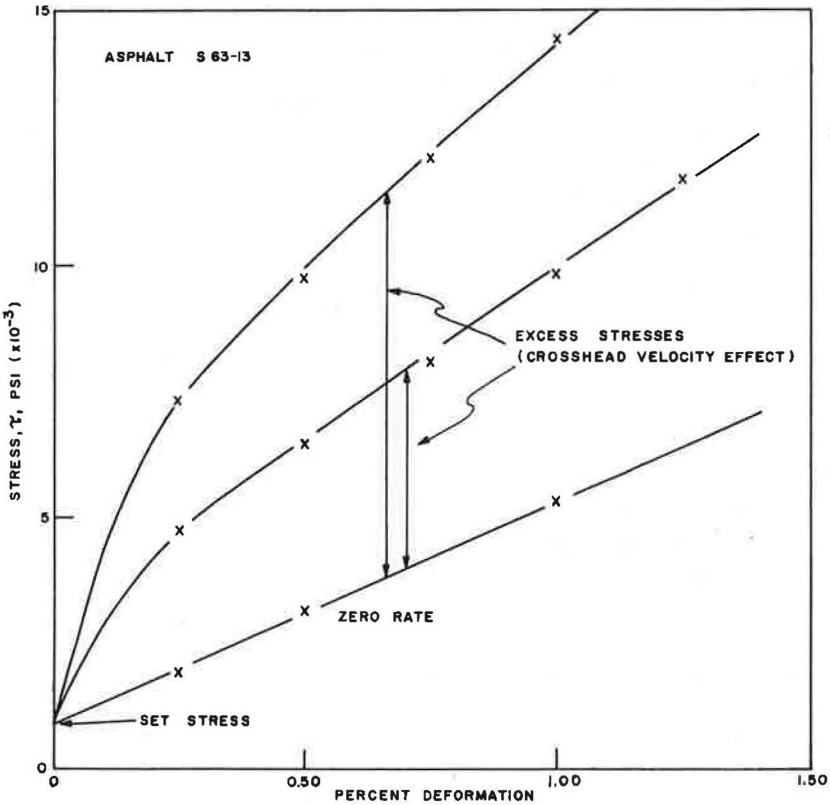

Figure 8. Normalized and corrected data for asphalt cement S63-13 with drag.

will have any influence (τ is a function of rate of strain). The effect of a given deformation in the past may thus be characterized by a time. This time is usually called the characteristic time and an interesting discussion of it has been presented by Slattery (13). Slattery explains the different viewpoints on characteristic times by Truesdale (17). Essentially, Slattery's contention is that differentiation between the contribution of the material and the contribution of the process itself to the overall time-dependence is difficult and somewhat arbitrary. Determination of different time constants when testing different materials under the same experimental conditions is enough to warrant assignment of these time constants to the materials.

[A Stokesian fluid is one where the stress caused by deformation is only a function of the rate of deformation; it may be Newtonian or non-Newtonian (14). Asphaltic cements used in paving have memories at ambient temperatures that will be between the limits for these two extremes because they are viscoelastic. These memory effects are indicated by the experimental curves of Figure 7.]

Conceptually, the relationships discussed for the dynamic stress at essentially a constant rate of deformation might be considered as follows: The time-varying stress resulting from the application of a constant rate of deformation will be primarily determined by the consistency of the material. Mathematically this dynamic stress is the product of three factors. It should be emphasized that this equation is of no rheological value unless K can be described at each material point and time.

$$\tau = MU \cdot K \cdot g(\text{memory}) \tag{1}$$

where

τ = dynamic stress above that for zero-rate deformation, dynes/cm^2;
MU = consistency modulus, poises;
K = rate of deformation = f(rate of compression and geometry); and
g(memory) = dimensionless time-dependent factor representing the memory of the material.

For the special cases of perfectly elastic material and Stokesian fluids, g = 0, and g = a constant independent of time, respectively.

Deformation With Lubrication

To eliminate the drag effect, the barrel of the cylinder and the plunger were coated with a thin film of silicone lubricant and compression tests carried out as described previously at 32 F. With the drag on the walls eliminated, the deformation during compression is homogeneous, allowing an invariant evaluation of the phenomenon. The parameters thus obtained when studying the response to different deformation histories can be evaluated without influence of different geometries and machine characteristics. A homogeneous compression occurs when three material particles remain colinear throughout the deformation. A simple check on the degree of homogeneity of the deformation is to deform different volumes of sample (V_1, V_2, etc.) and adjust the crosshead speed (K) such that

$$\frac{K}{V_1} = \frac{K}{V_2}$$

The response to these experiments should be identical.

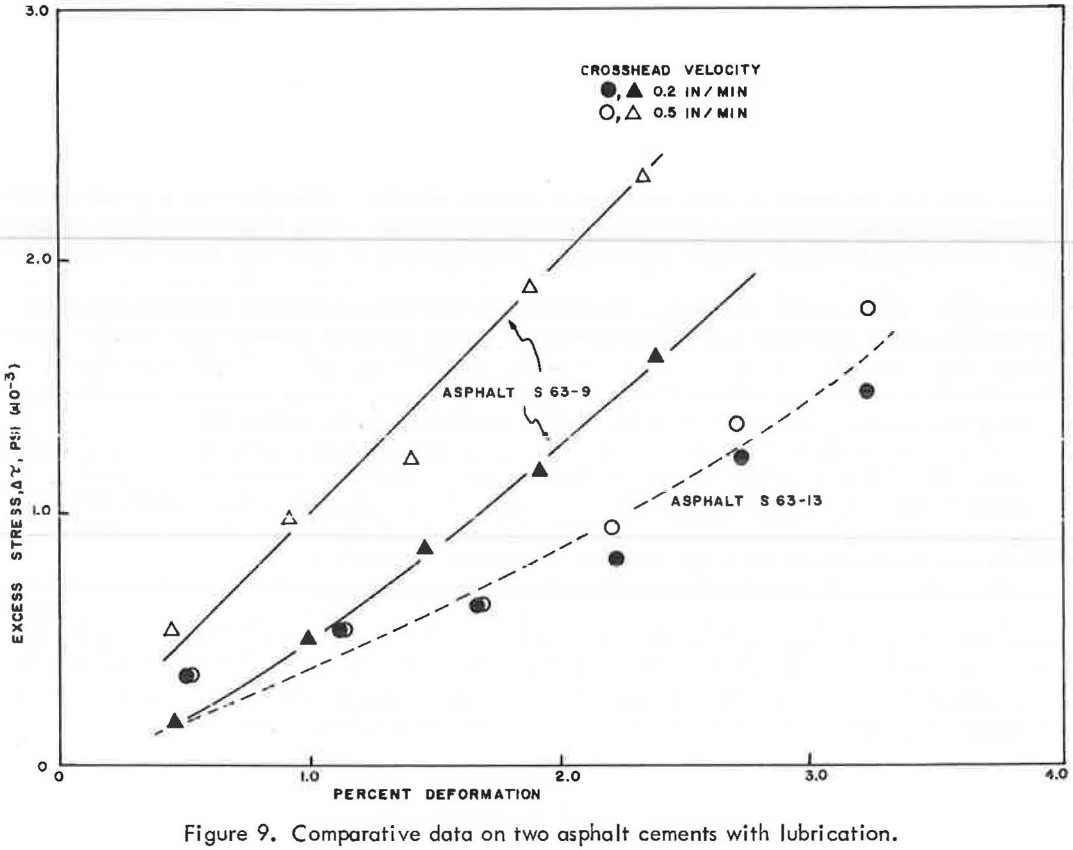

Figure 9. Comparative data on two asphalt cements with lubrication.

The results on two asphalts shown in Figure 9 present certain interesting information. Asphalt cement S63-9 is from a Texas source and it shows considerable difference in the deformation curves at two different rates of deformation. If the material were completely elastic, the stress developed would be essentially independent of the rate of deformation. This is approximated by the preliminary data for asphalt S63-13, which is an air-blown asphalt. Such materials are known to exhibit elastic properties with a decrease in viscosity at high rates of deformation.

In the case of asphalt S63-9, the excess stress above that for the zero deformation rate is a measure of visco-compression effects, which at this time have not been analyzed completely. However, the significant differences for the two asphalts in Figure 9 are considered important. This is particularly true because other information from work in this laboratory has indicated appreciable differences in the changes of their internal energies (u) with volume (v). This has been studied by Ronk, Busot, and Schweyer (20) in measuring the coefficients of thermal expansivity and isothermal compressibility. The value of $(\partial u/\partial v)_T$ can be evaluated, and it is an indication of the cohesive forces for these materials.

The basis for further consideration of predictions and correlations of the properties discussed may be found in Bondi (21) and Middleman (15). These references review most of the pertinent theories and correlation methods. However, the experimental observations reported in this paper cannot be explained rigorously by present theories. Most viscosity theories are limited by the assumption of incompressibility, or by considering deformations at constant density (isochoric shear and elongational experiments). Free-volume theories on the other hand have failed to rigorously explain the low-temperature viscosity data of certain liquids (22). Moreover, asphalts are complex mixtures requiring perhaps different and sophisticated mixture theories for each property considered.

SIGNIFICANCE OF RESULTS

Although considerable experimental data have been obtained, no quantitative measurements of deformation parameters are given for the preliminary homogeneous compressive deformation reported here. The significance of these results has not been completely developed as yet, and until this is finished it would appear that discretion should be employed before drawing conclusions. The difference in response between the "homogeneous" and "drag" compression tests will not be interpreted, but instead Table 1, giving empirical characteristic time, consistency, and compressibility, is presented.

The stress decay curves (Fig. 5) were used to evaluate a characteristic time. The ratio of the initial slope $(d\tau/dt)_{t=0}$ to the initial value of the rate of change of this slope $(d^2\tau/dt^2)_{t=0}$ has the dimension of time. This arbitrary time, when obtained at exactly the same experimental condition for different asphalts, is assigned as their characteristic time, θ. This is only an empirical indication of how long the asphalts are able to remember past deformations. Similarly, the excess stress at 2 percent

TABLE 1

PRELIMINARY EVALUATION OF MATERIAL RHEOLOGICAL PARAMETERS AT 32 F

Identification	Penetration 77 F	Viscosity 140 F, poises	Compressibility, percent strain/psi ($\times 10^4$)	Consistency μ, poises ($\times 10^{-12}$)	Characteristic time, θ, sec
Texas, SR Int. S63-9	85	1704	3.10	1.70	48
AB. Naphthenic S63-13	89	1726	3.74	0.74	32
Gulf Coast Naphthenic, SR S63-12	85	1368	3.10	2.53	84
Los Angeles basin S63-20	89	1109	2.85	2.66	82

deformation divided by a specific rate of deformation gives a measurement of consistency μ. The elastic compressibility was obtained from the stress-strain relation at zero rate of strain.

Many experiments have been carried out in the area of this research, but it is not the purpose here to present all the results because much of the exploratory work on repeatability, reproducibility, etc., will be discarded when procedures have become firm.

This information is presented in a preliminary fashion to indicate a new approach that might be taken for analysis of the rheological properties of asphalt. It represents an effort to obtain a better understanding in bridging the gap between the knowledge of the constitutive equations that define the material and utilization of this information in the actual design of a flexible pavement. The procedures and results as presented here are not considered final but are considered of sufficient interest to paving technologists to warrant presentation at this time and to solicit criticism and suggestions for additional work. This work is continuing and it is hoped that future information can be presented that will be more informative for application to design.

ACKNOWLEDGMENT

The assistance of Donald Ronk in preparing the drawings and certain aspects of the experimental work in this research is gratefully acknowledged.

REFERENCES

1. Hoiberg, A. J. (Editor). Bituminous Materials: Asphalts, Tars, and Pitches. Vol. 1. John Wiley and Sons, New York, 1964.
2. Mack, C. Theory of the Deformation Mechanism and Bearing Strength of Bituminous Pavements. Proc. AAPT, Vol. 23, p. 338, 1954.
3. Mack, C. A Quantitative Approach to the Measurement of the Bearing Strength of Road Structures. Proc. AAPT, Vol. 16, p. 264, 1947.
4. Shoor, S. K., Majidzadeh, K., and Schweyer, H. E. Rheology of Asphalt—A Set of Four Technical Papers: Nos. 340, 372, 373, 374. Florida Eng. and Indus. Expt. Sta., 1966.
5. Heukelom, W. Observations on the Rheology and Fracture of Bitumens and Asphalt. Proc. AAPT, Vol. 35, p. 358, 1966.
6. Schmidt, R. J., and Santucci, L. E. A Practical Method for Determining the Glass Transition Temperature of Asphalts and Calculation of Their Low Temperature Viscosities. Proc. AAPT, Vol. 35, p. 61, 1966.
7. Sisko, A. W. Tensile Strength of Asphalt Films and Road Life. Highway Research Record 231, p. 62-67, 1968.
8. Majidzadeh, K., and Schweyer, H. E. Viscoelastic Response of Asphalts in the Vicinity of the Glass Transition Point. Proc. AAPT, Vol. 36, p. 80, 1967.
9. Moavenzadeh, F. Asphalt Fracture. Proc. AAPT, Vol. 36, p. 51, 1967.
10. Krchma, L. C. Asphalt Consistency Control and Characterization by Flow Temperature. Proc. AAPT, Vol. 36, p. 16, 1967.
11. Gaskins, F. H., Brodnyan, J. G., Phillippoff, W., and Thelen, E. The Rheology of Asphalt: II. Flow Characteristics of Asphalts. Trans. Soc. Rheol., Vol. 4, p. 265, 1960.
12. Brodnyan, J. G. Use of Rheological and Other Data in Asphalt Engineering Problems. HRB Bull. 192, p. 1, 1958.
13. Slattery, J. C. Dimensional Considerations in Viscoelastic Flows. AIChE. Jour., Vol. 14, p. 516, 1968.
14. Coleman, B. D., Markovitz, H., and Noll, W. Viscometric Flows of Non-Newtonian Fluids. Springer-Verlag, New York, 1966.
15. Middleman, S. The Flow of High Polymers. Interscience Publ., New York, 1968.
16. Truesdale, C. The Elements of Continuum Mechanics. Springer-Verlag, New York, 1965.
17. Truesdale, C. The Natural Time of a Viscoelastic Fluid: Its Significance and Measurement. The Physics of Fluids, Vol. 7, p. 1134, 1964.

18. Lodge, A. S. Elastic Liquids. Academic Press, New York, 1964.
19. Majidzadeh, K. Durability Characteristics of Asphaltic Materials. Transportation and Research Center Rept. No. EES 300-1, Ohio State Univ., 1968.
20. Ronk, D. E., Busot, J. C., and Schweyer, H. E. The Compressibility and Expansivity of Asphalts. (To be published.)
21. Bondi, A. Physical Properties of Molecular Crystals, Liquids, and Glasses. John Wiley and Sons, New York, 1968.
22. Barlow, A. J., et al. Proc. Roy. Soc. (London), Ser. A, No. 292, p. 322, 1966.