# Rheology of Bitumens and the Parallel Plate Microviscometer

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The viscoelastic behavior of bitumens varies with film thickness. Therefore, for correlation of laboratory testing with field performance in bituminous pavements, it is important to match the geometry of testing to geometry of end use. The parallel plate microviscometer is well suited for this work, but the total displacement suggested by previous authors for most paving asphalts is too low.

Rotating parallel plates have been used to extend the useful range of the microviscometer.

● IT was introduced to the bituminous field in 1954 by Labout and Van Oort (2, 4, 5) Griffin et al. (1) simplified the thin film preparation.

The Naugatuck Chemical Division's work began before a commercial instrument was available so the Division designed and built its own. Inasmuch as the heart of the instrument, the glass plate assembly, is identical with that of previous authors, the instrument is described only as an example of an inexpensive unit which is both versatile and precise.

The parallel plate microviscometer is an important contribution to the bituminous field and especially to the paving field, not only because it is a fundamental instrument, but because it is perhaps the only instrument in which thin films, duplicating the binder thickness in a pavement, can be studied. In bituminous concrete pavements, the film thickness of the asphalt binder is of the order of 5-10 microns (3). This is vastly different from the film thickness encountered in most instruments used for viscosity measurement.

It has been reported by the previous authors that the physical behavior of bitumens does not change with film thickness which would make the latter advantage of the microviscometer unimportant. However, the authors have found that viscoelastic behavior does change with film thickness and herein lies the real advantage of the microviscometer. In addition, the authors have found that for most paving asphalts, the suggested displacement of 200 microns (1) of the upper plate for each shearing stress is insufficient.

## EXPERIMENTAL DETAILS

### Apparatus

The microviscometer is shown in Figure 1. Movement of the upper plate is followed with a microscope, 600 X, focused on a stage micrometer 2 mm in length, graduated in units of 0.01 mm. The level of the water bath is adjusted to be above the surface of the top plate and below the string.

### Film Preparation

The film preparation step which has already been described by previous authors requires considerable practice, but is made easier by using the film preparation unit shown in Figure 2. To prepare a uniform film, the hot glass plates-binder sandwich is placed in a template on the unit and pressed to the desired thickness with the aid of a 3/8- x 2- x 2-in. piece of glass over the top

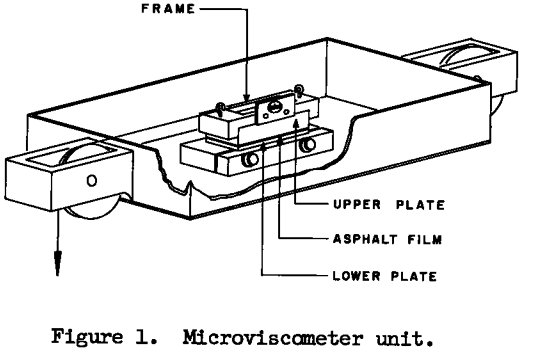



Figure 1. Microviscometer unit.

glass plate. This piece of glass keeps the fingers out of the way for viewing and from being burnt.

Application

Displacement of the top glass plate is plotted with respect to time on rectangular graph paper. Viscosity is calculated as follows:
Viscosity is calculated as follows:

$$\text{Viscosity in poises} = \frac{\text{Shearing Stress}}{\text{Rate of Shear}} = \frac{\dfrac{\text{Load in grams x 980}}{\text{Area in cm}^2}}{\dfrac{\text{Displacement in cm}}{\text{Film thickness in cm x seconds}}}$$

## DISCUSSION OF DATA

Curves obtained using a 127 penetration Californian asphalt are shown in Figure 3. This Californian asphalt is showing Newtonian behavior, constant viscosity at varying rates of shear, under these conditions of testing as shown in Figure 4 (Curve 1).

All the curves in Figure 3 are straight lines passing through the origin which is typical for Newtonian fluids. If it had been known in advance that this asphalt had Newtonian behavior under these conditions, it would only have been necessary to obtain one curve in Figure 3 in order to plot the curve in Figure 4. The instrument is sensitive enough to obtain an accurate curve for Newtonian materials after only 25 to 50 microns displacement. Curves No. 2 and 3 (Fig. 3) then could have been completed in 2 and 10 min, respectively.

Results obtained using an 85 penetration Venezuelan asphalt are shown in Figure 5. In a viscoelastic material, the rate of shear under a given load may be rapid initially, but then decreases to a constant value. This condition is visible in Curves 5 and 6 (Fig. 5). The steady state viscosity is calculated from the straight-line portion of these curves. These results plotted as viscosities in Figure 4 (Curve 2) indicate that this asphalt has viscoelastic behavior, viscosity decreasing with increasing rate of shear.

For the Californian asphalt, a displacement of only 50 microns was sufficient. For the Venezuelan asphalt, the straight-line portion of Curve 5 (Fig 5), was reached at a displacement of 80 microns and after 4 min of testing.

Data obtained with the same Venezuelan asphalt at 140 F are shown in Figure 6. In Curve 9, the straight-line portion was reached in 25 min at a displacement of 400 microns. In Curve 10, it was reached in 45 min at 300 microns. The true viscosity rate of shear curve for this asphalt at 140 F is shown in Figure 4 (Curve 3). A flase viscosity curve is also shown in Figure 4 (Curve 4) which results from plotting the curves in Figure 6 after only 100 microns of displacement. The initial 100 micron portions of these curves appear as straight lines.

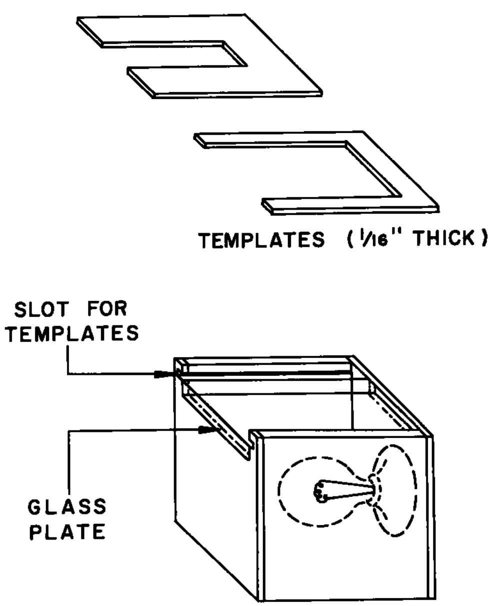


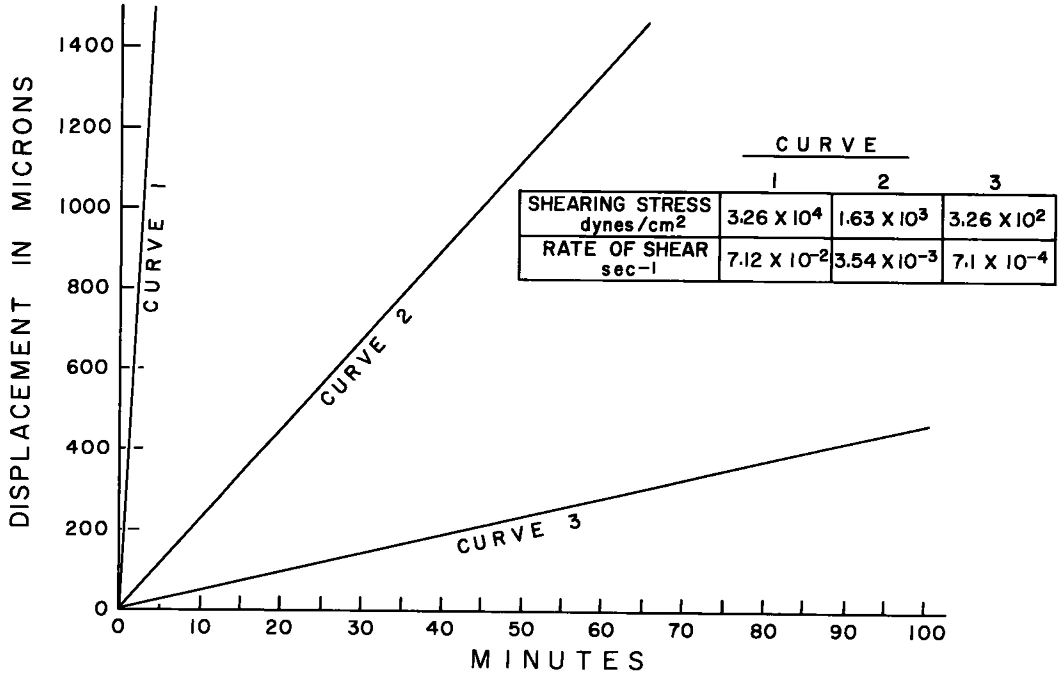

Figure 2. Film preparation unit.

These three curves were taken to 0.15 cm displacement to be sure that no further change in rate of shear occurred.

Nine paving grade Venezuelan asphalts obtained from the same supplier and which met identical specifications (that is, penetration, softening point, ductility, etc.) were studied over a wide range of test conditions.

None were close enough to Newtonian behavior over a practical temperature and rate of shear range to allow the true steady state viscosity behavior to be determined from curves of less than 400 microns of displacement.

After similar studies on many other types of paving asphalts including Smackover,




| | CURVE 1 | CURVE 2 | CURVE 3 |
|---|---|---|---|
| SHEARING STRESS dynes/cm$^2$ | $3.26 \times 10^4$ | $1.63 \times 10^3$ | $3.26 \times 10^2$ |
| RATE OF SHEAR sec$^{-1}$ | $7.12 \times 10^{-2}$ | $3.54 \times 10^{-3}$ | $7.1 \times 10^{-4}$ |

Figure 3. Displacement-time curves at 77 F for Californian asphalt, 127 penetration, 108 micron film.

Wyoming, Arkansa, Santa Maria, Martinez, Midcontinent, Texas, two from Canada, and others, the authors conclude that probably less than 10 percent of the paving asphalts should be evaluated with a displacement less than 400 microns.

Asphalts which deviate widely from Newtonian behavior present more of a problem. An example is the air blown Midcontinent asphalt, 135 pen. 127 F softening point, for which data are shown in Figure 7. In Curves 11, 12 and 13, the initial rate of shear is high, then it decreases as though it would reach a constant value; but instead, due to structural breakdown of the rheological units in the asphalt, it begins to increase again. In Curve 13, the rate of shear becomes constant in about 1 hr after 400 microns of displacement. In Curve 12, a constant rate of shear was not obtained until after 700 microns. In Curve 11, the initial bending due to elastic response and the opposite bending due to structural breakdown has all blended into a straight line. In Curve 14, no upward bending of the curve occurred after the straight-line portion was reached.

The curve obtained using a rubberized asphalt joint sealing material is shown in Figure 8. In this case no straight line portion has been reached even at 1,800 microns.

The same precautions must be followed when studying tars.

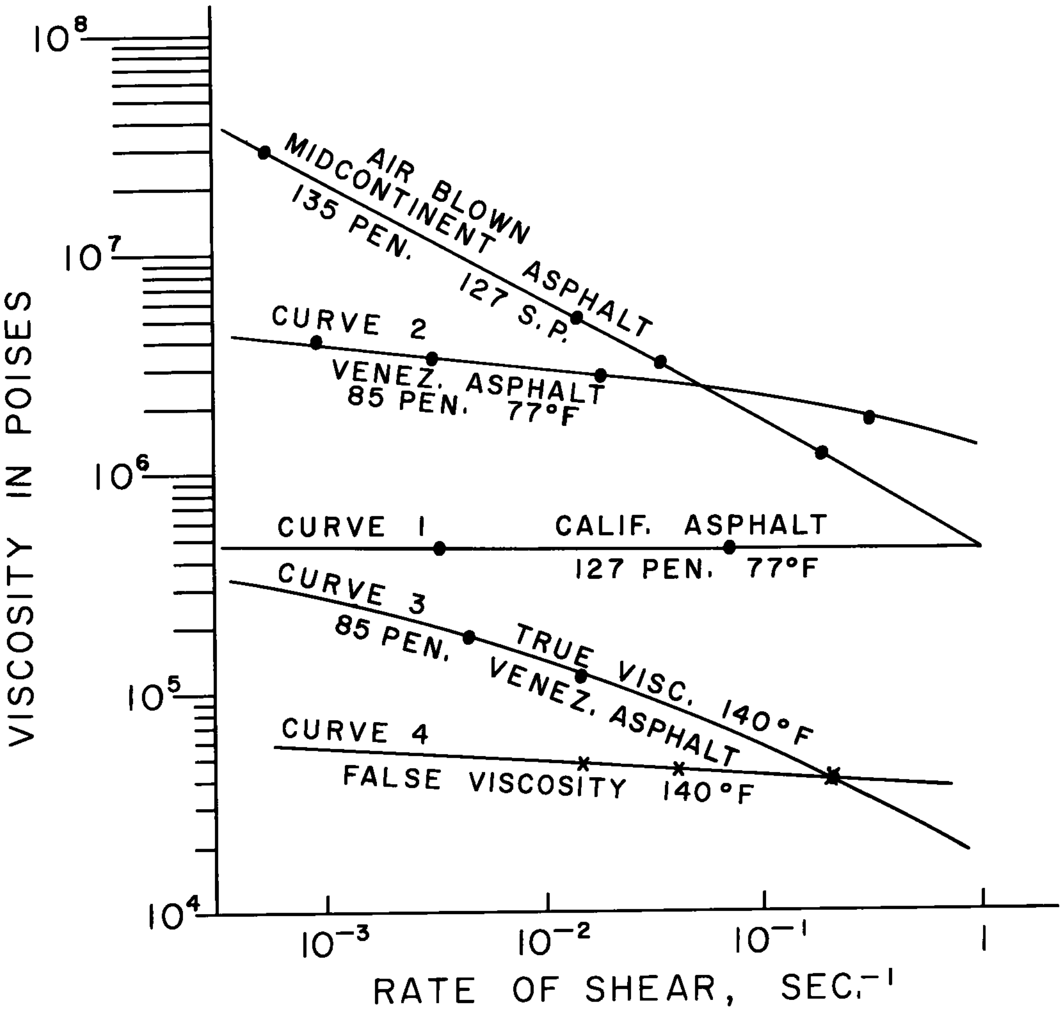



Figure 4. Viscosity at varying rates of shear.

## Variation of Viscoelastic Behavior with Film Thickness

Previous authors have indicated that the viscoelastic behavior of bitumens could be studied in the parallel plate microviscometer without particular attention to film thickness within the range of the instrument (that is, 10 to 100 microns), because the behavior was not dependent on film thickness.

The authors find that viscoelastic properties change with film thickness and have reported it elsewhere (7).

The reason for the two points of view on this subject may be due to the displacement-time factor.

Typical displacement-time curves for various film thicknesses are shown in Figure 9. Notice that more than 100 microns of displacement was necessary to reach the straight-line portion in Curves 16, 17 and 18. A total testing time of about 3 hr was necessary to be sure that the straight line portion of Curve 18 was reached.

When the slopes 1, 2, 3 and 4 taken from the straight-line portion of each curve were used to calculate the viscosity, Curve 1 in Figure 10 was obtained which clearly shows the dependency of viscosity on film thickness.

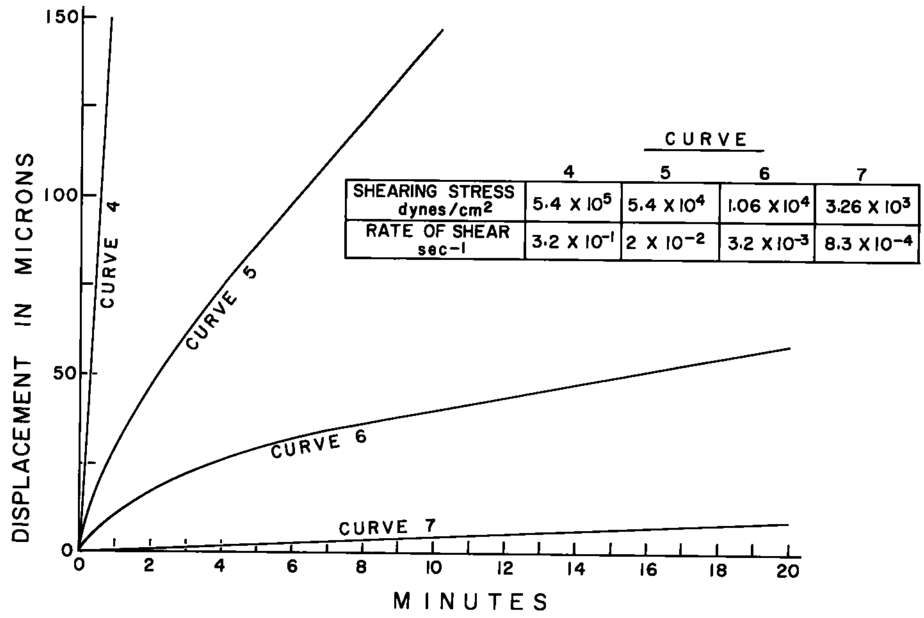



Figure 5. Displacement-time curves at 77 F for Venezuelan asphalt, 85 penetration, 10 micron film.

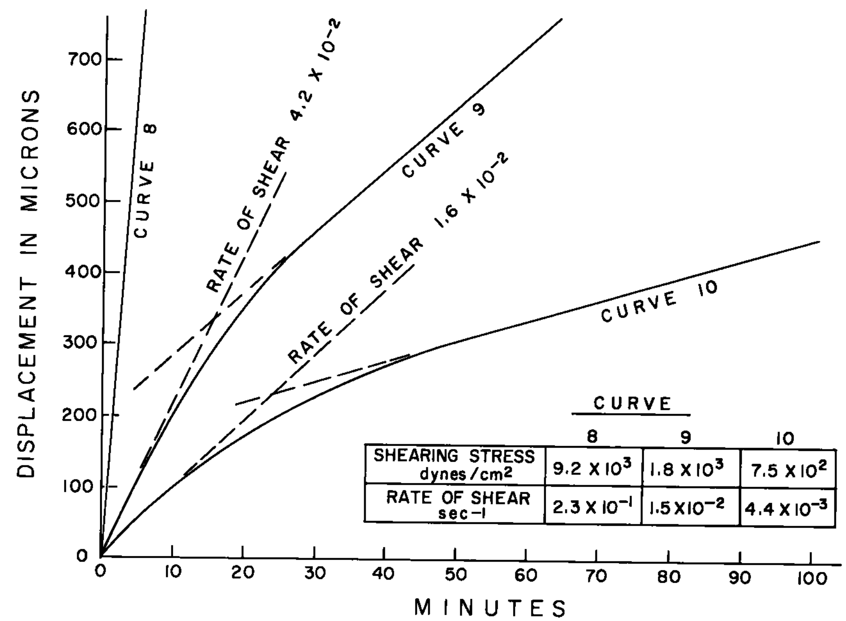



Figure 6. Displacement-time curves at 140 F for Venezuelan asphalt, 85 penetration, 10 micron film.

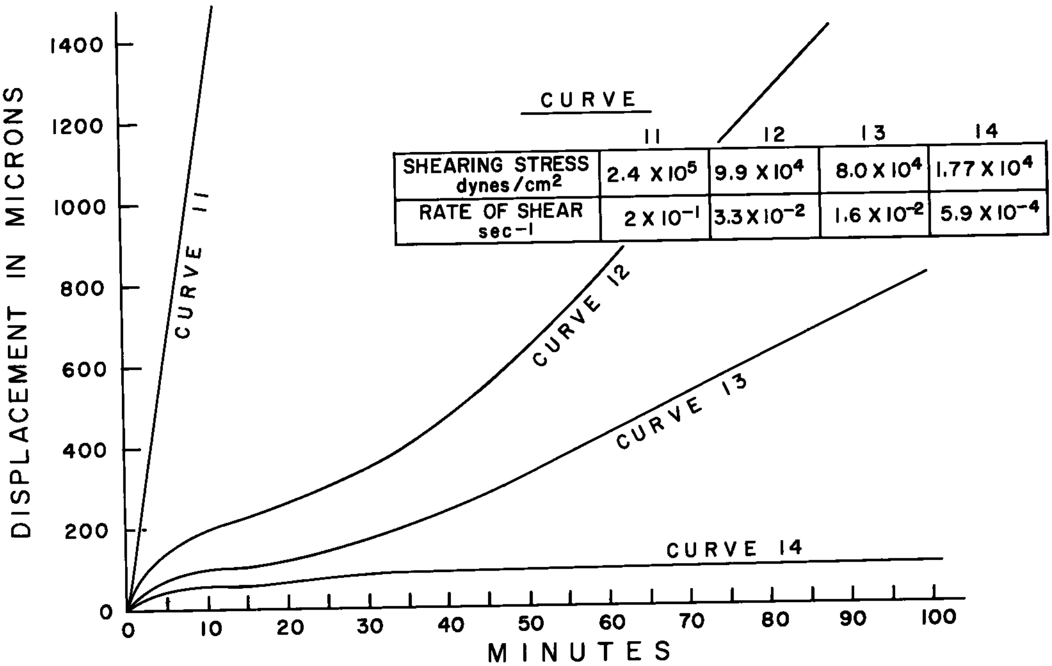



Figure 7. Displacement-time curves at 77 F for Midcontinent air blown asphalt, 135 penetration, 127 soft point, 10 micron film.

When slopes 1, 5, 6 and 7, taken from the first 100 micron displacement and which also appear to be straight lines, were used to calculate the viscosities, Curve 2 in Figure 10 was obtained which indicates that viscosity does not change with film thickness.

It is not surprising that the viscosity under shear increases with decrease in film thickness because it has been known for some time that tensile increases in the same manner. This was recently reported by Mack (3) and Wood (7). This work can be easily duplicated using the same 2-cm x 3-cm x ¼-in. glass plates used in the parallel plate microviscometer.

A circular film of any desired thickness is prepared between crossed plates as shown in Figure 11. For films under 10 microns thickness, the diameter of the circle should not be greater than 1 cm. The advantage of using glass plates instead of metal is that uniformity of the film (that is, thickness and lack of cavities) can be insured by observation. A jig to hold the tensile specimen is also shown in Figure 11. The jig can be placed in a temperature controlled bath on the tensile machine. A compression head is used on the tensile machine because the jig reverses the force to one of tensile on the specimen.

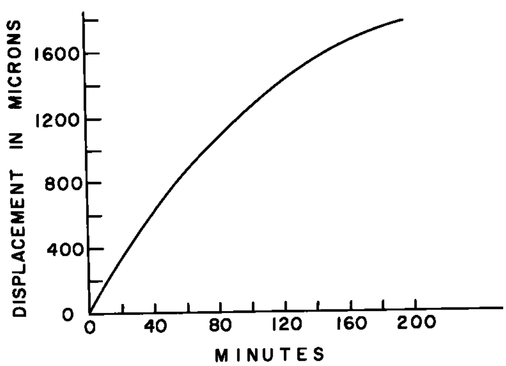



Figure 8. Displacement-time curve at 77 F for rubberized asphalt joint sealer, 10 micron film.

The effect of film thickness on tensile for the same 110 penetration asphalt is shown in Figure 12.

Notice that in Figures 10 and 12 this asphalt under these conditions of testing has what can be called an infinite thickness region, above which the viscosity or tensile does not change.

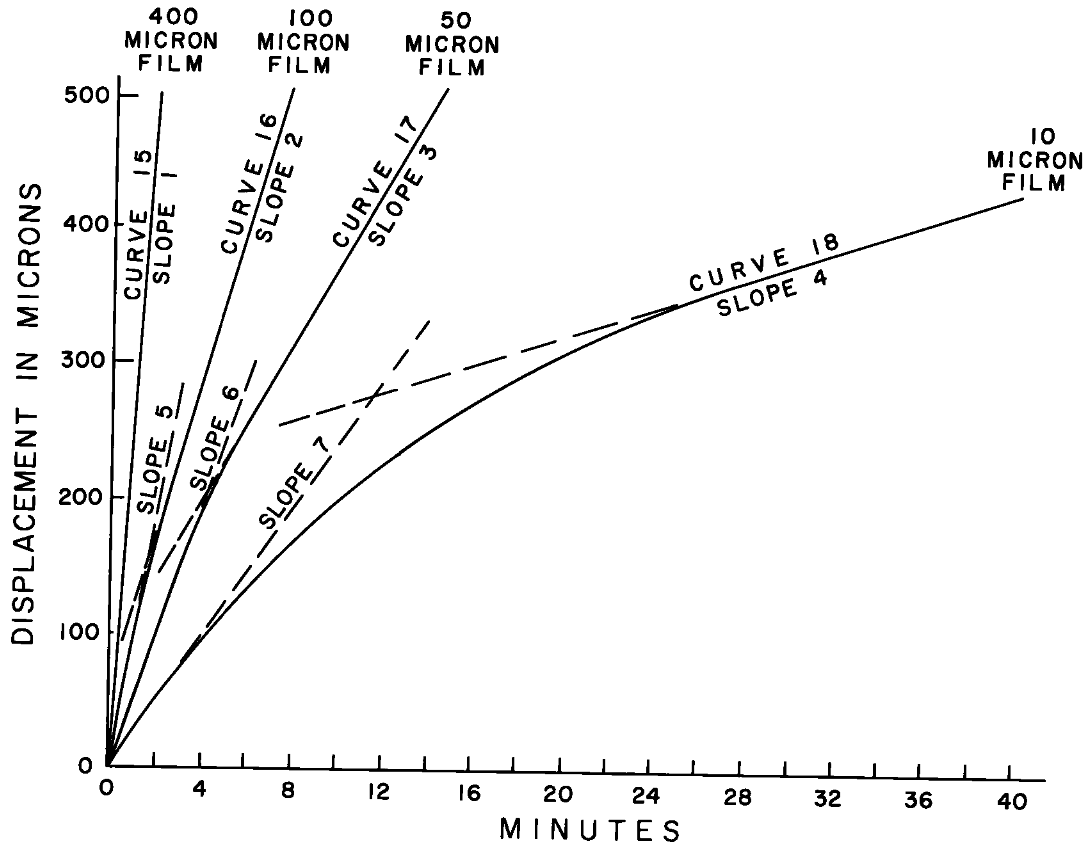



Figure 9. Displacement-time curves at 77 F and various film thicknesses for Venezuelan asphalt, 110 penetration.

Viscosity measurements on this asphalt were made in many types of fundamental viscometers such as capillary, rotational cylinder and disc, falling concentric cylinder, etc., up to asphalt thicknesses of ¼ in. (6,350 microns) and within experimental error, the viscosity was the same as the viscosity in the infinitely thick region in Figure 10.

This also applies to tensile data on films ¼ in. thick.

It is unlikely that the change in properties with film thickness in films greater than one micron thick can be attributed to long range London-vander Waal forces (6).

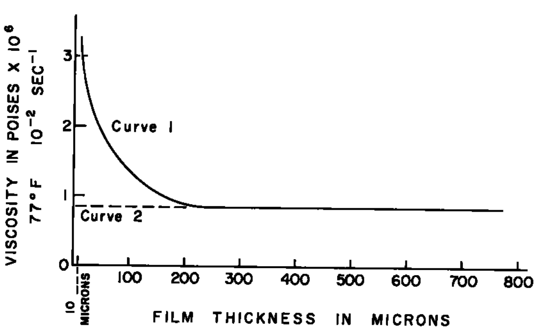
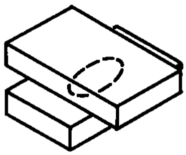



Figure 10. Effect of film thickness on viscosity at 77 F of Venezuelan asphalt, 110 penetration.

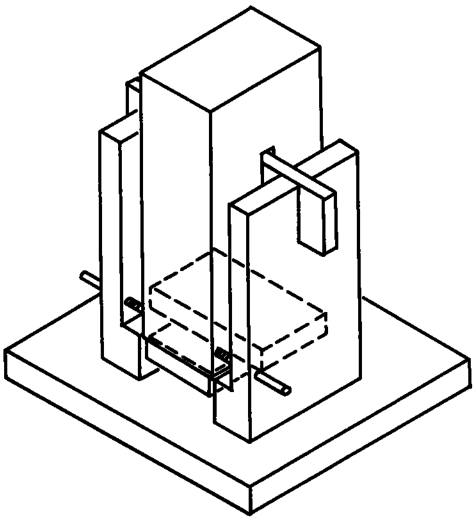
The answer probably lies in the degree and rate of structural breakdown in the bitumen. Any colloidal material which exhibits thixotropy must pass through intermediate stages of breakdown.

The straight-line portion, constant rate of shear, of the displacement-time curves represents an equilibrium between structural breakdown and reformation.

It has been shown that in thin films, the elastic effect on flow properties in many cases is not overcome until the displacement is many times the thickness. The true steady state viscosity of a 10



TENSILE SPECIMEN



TENSILE SPECIMEN HOLDER

Figure 11. Tensile specimen and holder.

micron film of rubberized asphalt (Fig. 8) was not reached in 1,800 microns of displacement.

In thick films such as occur in many viscometers, limitations of the particular type of viscometer may not allow the necessary amount of rate of displacement to take place to completely overcome initial elastic effects on flow or to reach the upward swing of displacement-time curve if one is present.

<u>Rotational Parallel Plate Microviscometer</u>

The present rectangular glass plates limit the amount of displacement to 0.15 cm, 5 percent of the total length. Actually the limit is 0.30 cm if the test is started with the top plate already off-set 0.15 cm. Even 0.30 cm is not sufficient for highly viscoelastic materials or thick films of even moderately viscoelastic materials under certain conditions of testing. The authors have overcome this by using circular glass plates which are rotated. In many ways the rotational movement lends itself to easier ocular or automatic measurement.

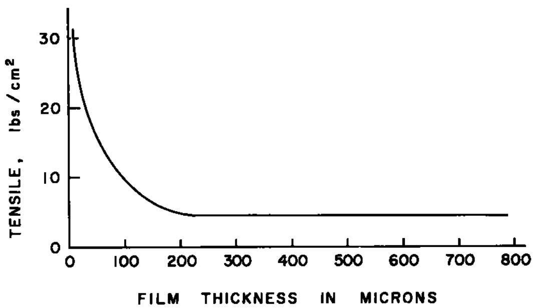



Figure 12. Effect of film thickness on tensile strength at 77 F of Venezuelan asphalt, 110 penetration.

## CONCLUSIONS

The viscoelastic behavior of bitumens has been shown to vary with thickness when very thin films are involved. This must be taken into account especially in pavement work. The geometry of testing must match the geometry of end use.

The parallel plate microviscometer is an important contribution to the bituminous paving field because fundamental rheological data can be obtained on very thin films comparable to those obtained in practice.

Care must be taken in the use of the microviscometer that the total displacement is sufficient to reach a steady state condition of flow. This amount of displacement may be many times that suggested by previous authors.

The use of circular plates extends the useful range of the microviscometer.

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