# *Dynamic shear rheometers pave the way for quality asphalt binders*

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Rheological instruments and rheological measurements find applications in any industry where the flow characteristics of a material determine its processibility, performance, and/or consumer acceptance. Characterizing and understanding the rheological properties of asphalt binders in both the molten and solid state is fundamental in formulating the chemistry and predicting the end-use performance of these materials.

Rheology is the branch of science dealing with the flow and deformation of materials. The materials under investigation can range from low-viscosity fluids, semisolids, and polymer-modified asphalt to solid-state rigid samples. In the case of asphalt, rheological behavior is controlled directly by molecular structure, crystallinity, association crosslinking, and filler type and amount. Whether involved in manufacturing the asphalt, producing the machinery used in asphalt production, or processing the asphalt into a final product, rheological measurements are of practical importance. These quantitative, objective measurements bridge the gap between molecular structure, processibility, and ultimate performance properties.

Asphalt binders' behavior depends on temperature and time of loading. Unlike steel, which is considered a completely elastic material that can store energy indefinitely, asphalt binders cannot sustain a load without showing time-dependent deformation known as creep. Additionally, some of the input energy is dissipated in the material and results in a permanent set. This is called viscoelastic behavior. Samples exhibiting viscoelastic behavior are well suited for rheological characterization.

At high temperatures or long time of loading, asphalt binders act like high-viscosity flowable fluids. Viscosity is defined as the resistance to flow. As the viscosity decreases, a sample will flow more readily under the same conditions of temperature and load. At low temperatures or short loading times, asphalt binders act like elastic solids. Elastic solids approach the response of steel, where all the input energy is recoverable.

Additionally, there remains another unique characteristic of asphalt binders: environmental and/or chemical aging. Since asphalts are organic materials, they are susceptible to oxidation, chemical association, and physical hardening. All these phenomena cause changes in the asphalt binder with time. Rheology is a useful tool in quantifying these effects.

Traditional tests used to characterize asphalt binders were either penetration or simple one-point viscosity tests using a capillary device held at constant temperature. Both of these measurements have shortcomings and provide only limited insight into the overall flow behavior and ultimate end-use performance of an asphalt binder. Often these traditional tests provide misleading results in regard to the effect of additives, modifiers, and environmental conditions.

**Figure 1** *VISCOTECH DSR.*

In 1987, the Strategic Highway Research Program (SHRP) began developing new tests for measuring the properties of asphalt. The new tests measure physical properties that can be directly related to field performance by rigorous engineering principles. Since asphalt binder behavior depends on both time and temperature, the ideal test instrument would evaluate both of these variables. The VISCOTECH dynamic shear rheometer (DSR) (*Figure 1*), a research-grade DSR, and SPECTECH DSR (*Figure 2*), a specification-grade DSR (both from **ATS RheoSystems,** Bordentown, NJ), are capable of performing both of these tests. Both units feature dry heating/cooling of the test specimen. In a dry system, the test sample is not immersed in a fluid media to control the sample temperature. The dry system has many advantages such as response time, ease of use, ergonomics of sample edge trimming, and temperature range and stability.

## *Temperature sensitivity of asphalt binders*

Much has been written about the temperature sensitivity of asphalt binders. As a result, specification grading requires temperature control to ±0.1 °C.

**Figure 2** *SPECTECH DSR.*

*Figure 3* shows the response of a typical original asphalt binder at 64 °C as a function of time run with the VISCOTECH DSR. The data show the temperature stability and constant complex shear modulus G′ over 4000 sec.

Specification grading requires that the asphalt binder be evaluated at a variety of temperatures from 5 to 85 °C. Therefore, the time required for the DSR to change and then equilibrate the asphalt sample at the test temperature is paramount. *Figure 4* shows an original asphalt binder response when heated from 64 to 70 °C and *Figure 5* shows the same sample cooled from 64 to 58 °C. The important parameter is the time for the sample, not the instrument temperature controller, to reach thermal equilibrium.

The time to attain a steady response is greater in cooling mode than in heating mode. From the cool-

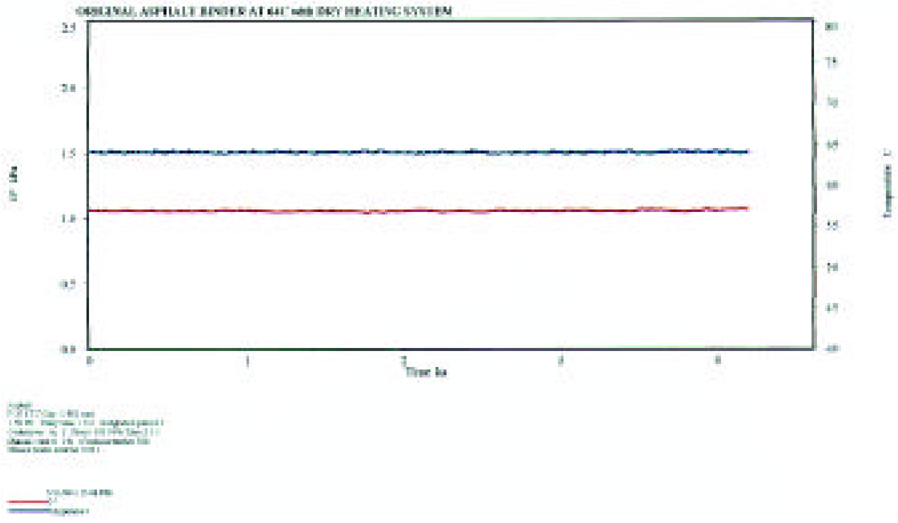

**Figure 3** *VISCOTECH DSR stability at 64 °C on an original asphalt binder.*

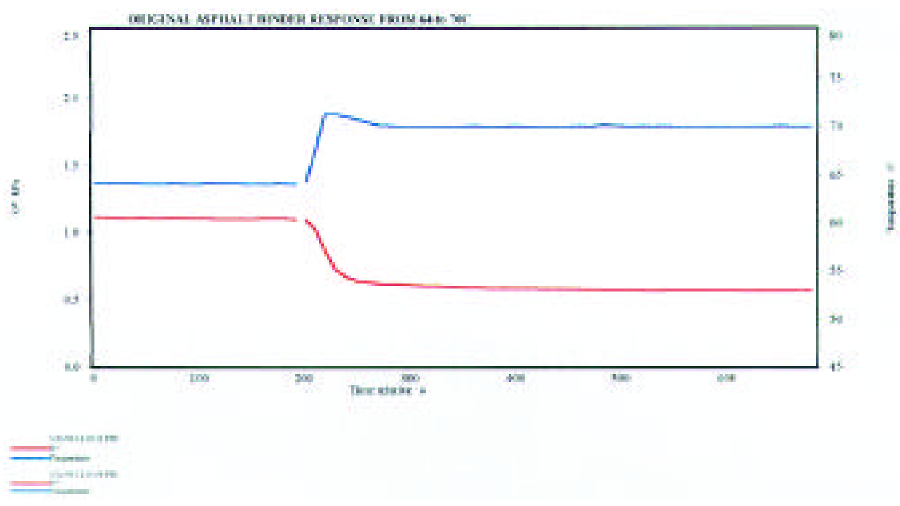

**Figure 4** *VISCOTECH DSR heating transient response 64–70 °C.*

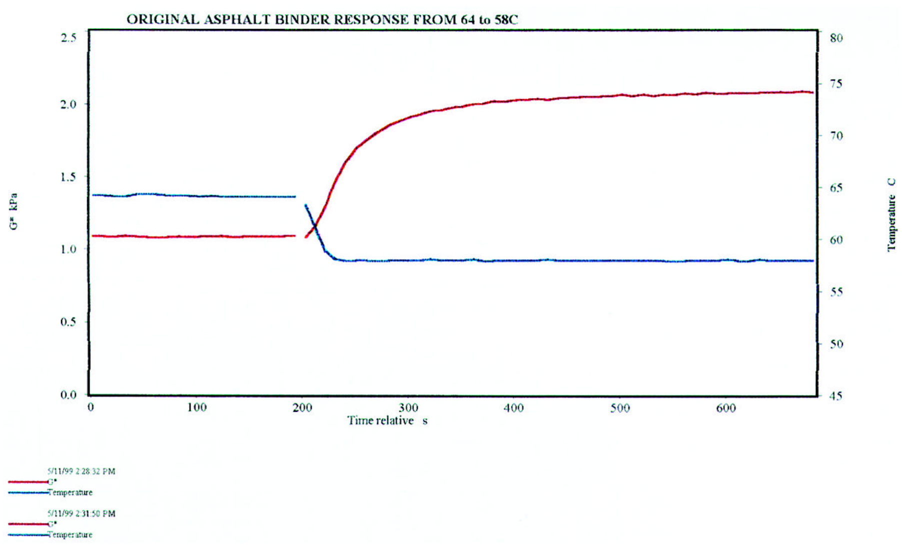


**Figure 5** *VISCOTECH DSR cooling transient response 64–58 °C.*

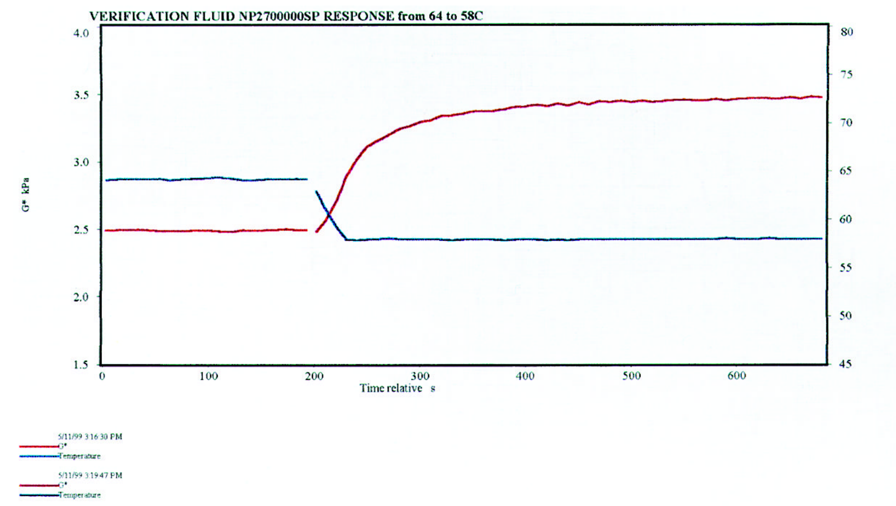


**Figure 6** *Temperature dependence of an original asphalt binder versus a calibration standard.*

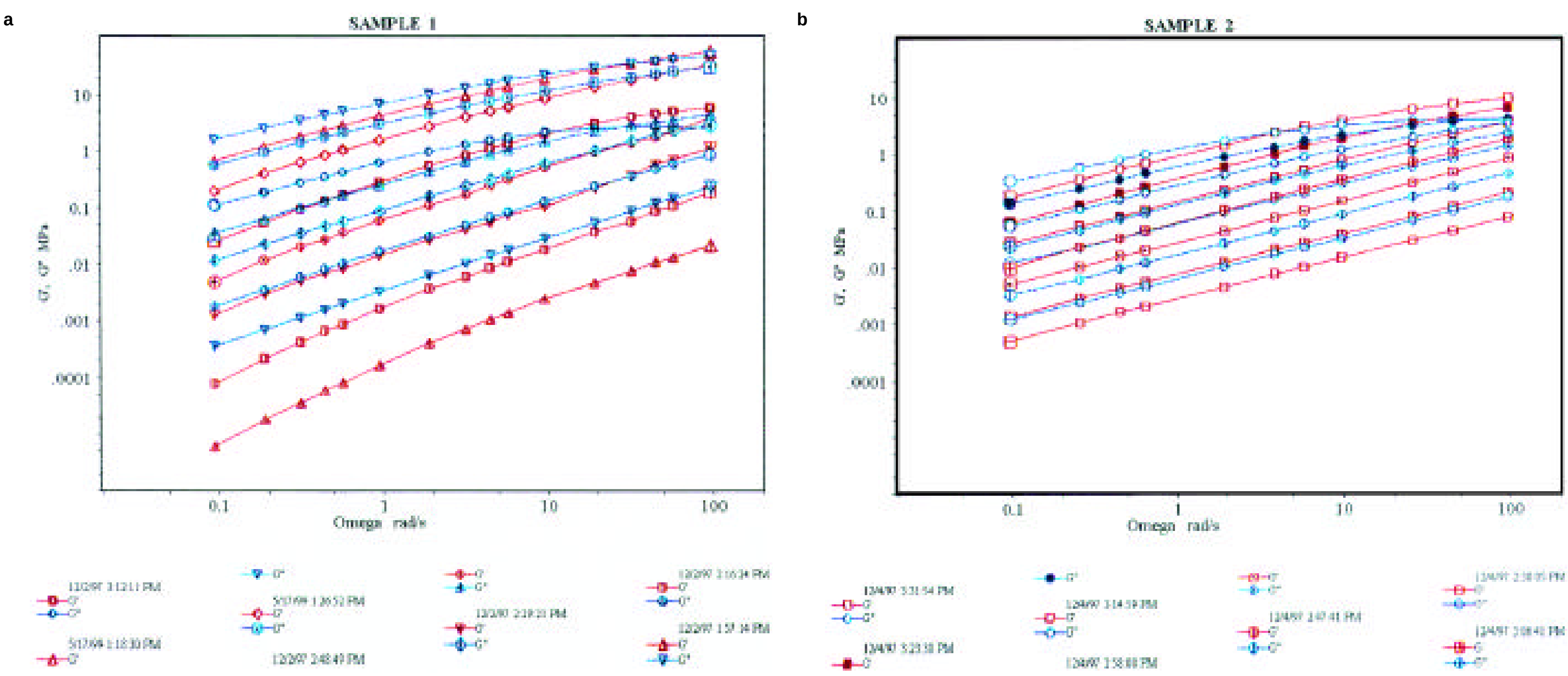


**Figure 7** *Shear modulus versus frequency results for samples 1 and 2 at selected temperatures.*

ing mode results, the sample has attained steady-state response after 5 min. In a comparable wet system, this time can exceed 30 min.

To determine the relative temperature sensitivity of an asphalt binder, a sample of DSR verification fluid N2700000SP was also run from 64 to 58 °C. The results are shown in *Figure 6*. The asphalt binder shows an approx. factor of 2 greater temperature dependence than the DSR verification fluid in this range.

### *Polymer-modified asphalt binder*

Polymers are often added to asphalt binders to improve the performance behavior with particular emphasis on obtaining desired viscoelastic properties. As an example, a polymer-modified asphalt demonstrates increased ring and ball softening point, reduced penetration, increased ductility, and increased toughness and tenacity. These effects become more pronounced with increasing polymer loading. Although extensive efforts have been directed at improving the quality and performance of asphalt paving materials, there appears to be no consensus among the various authorities concerning the least expensive and most effective methods to reduce or eliminate cracking, potholes, distortion, and other forms of pavement distress. Since the first sign of pavement failure is usually the formation of tiny, hairline cracks, it is particularly important to produce road surfaces that are tough and crack resistant, preferably through the use of additives that impart greater strength and extendability, especially at low temperatures when shrinkage forces and embrittlement are likely to promote failure.

In the past few years, much experience has been acquired in petrochemical research with regard to the rheology of polymers. At the same time, the technologies applied to the research of rheological characteristics of asphalt binders have been developed. This new research shows that asphalt binders can be characterized more rigorously with viscoelastic data.

### *Time–temperature superposition*

One of the primary analytical techniques used in analyzing the dynamic viscoelastic data for asphalt involves the construction of master curves for dynamic complex moduli, complex viscosity, and phase angle. In constructing such master curves, use is made of the time–temperature superposition principle. In constructing a master curve using time–temperature superposition, dynamic data are first collected over a range of temperatures and frequencies. A standard reference temperature must then be selected for the master curve. The data at all other temperatures are then shifted with respect to time until the curves merge into a single, smooth function. The time–temperature superposition principle also applies to materials undergoing a transition such as the glass transition.

In a master curve, one or more of the viscoelastic functions is plotted against frequency. At very high frequencies, the elastic modulus approaches a limiting value, which is the glassy state modulus. At low frequencies, the slope of a log–log plot of the elastic modulus versus frequency approaches 2, which signifies that viscous flow has been reached, and that the asphalt is behaving as a Newtonian fluid. Using the characteristic parameters of the master curve, along with the mathematical relationships for time–temperature dependence, it is possible to construct a unified mathematical model for the rheological behavior of asphalt binders.

Tests were performed on two asphalt binders in the temperature range from –10 to 50 °C in order to determine the most significant viscoelastic function. At temperatures from –10 to 35 °C, 8-mm-diam parallel plates were used in performing the dynamic testing. Shear moduli (G′, G″) and complex viscosity (η*) are plotted versus frequency (rad/sec) in *Figure 7a* and *b* and *Figure 8a* and *b*, respectively. The complex viscosity (η*) of sample 2 increases considerably with decreasing frequencies. On the other hand, sample 1 exhibits Newtonian behavior in the region of lower frequencies. Unmodified paving asphalt usually exhibits Newtonian flow behavior at temperatures in excess of 50 °C.

*Figures 9* and *10* are master curves using the Williams, Landel, and Ferry (WLF) shifting procedure in the temperature range of –10 to 50 °C for samples 1 and 2, respectively. In this case, the reference temperature selected was 25 °C. Sample 2, containing 5% styrene butadiene rubber (SBR), has a

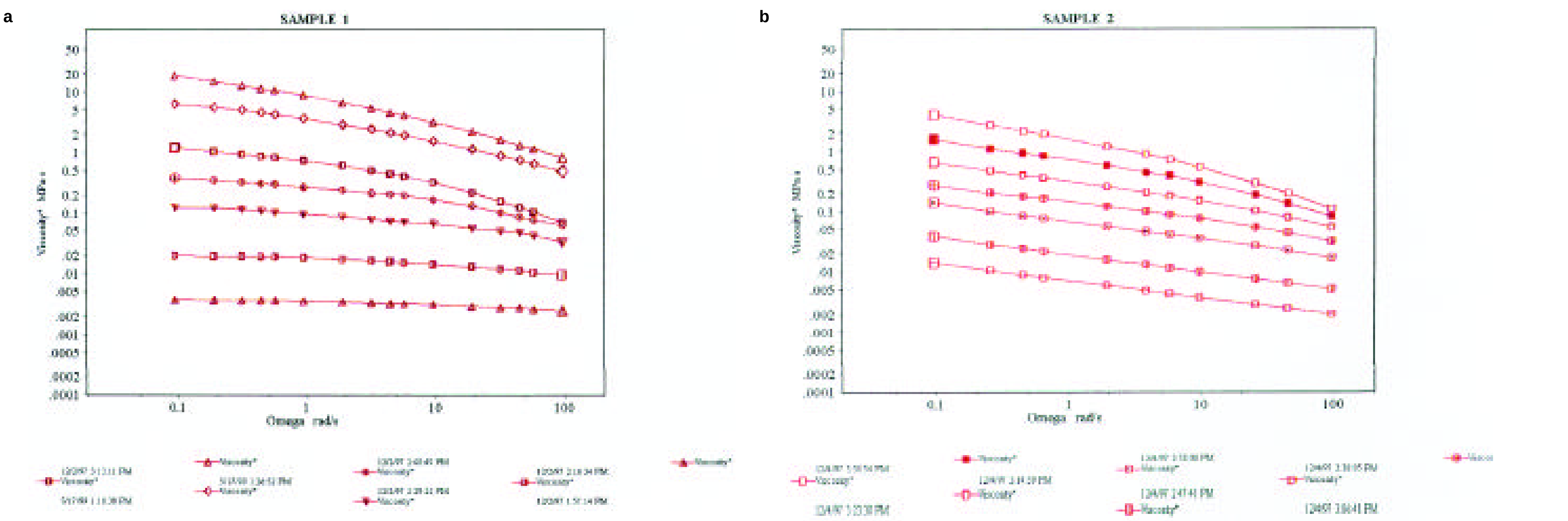


**Figure 8** *Complex viscosity versus frequency results for samples 1 and 2 at selected temperatures.*

crossover point of G′ and G″, very close to sample 1. However, in the terminal zone, sample 1 approaches Newtonian behavior with a phase angle near 90°, indicating a constant viscosity at long times, while sample 2 levels off at 65°, indicating an increasing viscosity at long times. The addition of a small amount of SBR results in a dramatic effect on the rheological properties and ultimate end-use performance characteristics of this asphalt binder in the long time, i.e., low rates of deformation and application areas such as rutting.

## *Creep tests*

Creep tests give extremely important practical information and, at the same time, useful data to one interested in the theory of the mechanical properties of asphalt binders. Since asphalt pavements are designed to be flexible, they must quickly return to their original configuration after loading. Rutting, pushing, and shoving are just a few of the failure mechanisms associated with inelastic or permanent deformation. Repeated loading without complete binder recovery is also a cause of fatigue cracking. Although the quality and gradation of the aggregate are important parts of the asphalt mix performance, the creep response of the binder is also a contributing factor. As creep is a time-dependent function, it is necessary to monitor recovery per unit time or to stipulate a time interval for an expected recovery. An important measure of polymer performance is its ability to recover after deformation. Since asphalt pavements are designed to be flexible, they must quickly return to their original configuration after loading. *Figure 11* illustrates a creep/recovery test at a constant stress of 1000 Pa applied over a time of 500 sec and recovery time of 1000 sec at 25 °C for samples 1 and 2. The results indicate that sample 2 creeps less and recovers more slowly than sample 1. As mentioned above, these data indicate that sample 2 is a stiffer binder with superior long-time creep properties.

## *Rheometer setup*

The VISCOTECH DSR and SPECTECH DSR are supplied standard will all measuring systems and software to perform asphalt binder testing according to AASHTO TP5-97 specification. The base-model VISCOTECH rheometer is a modular product with a wide range of measuring systems and accessories. Additional measuring systems for VISCOTECH are concentric cylinders, cone/plate, parallel plate, double concentric cylinders, closed/pressure cells, and dynamic mechanical analysis (DMA). Special measuring systems for low volume, high shear rates, and high sensitivity are also available. The measuring geometries can be made in stainless steel, titanium, polycarbonate, or any user-defined material. The instrument includes normal force capability for reproducible sample loading history, thermal expansion measurements, and can be configured with a patented Differential Pressure Quantitative Normal Force Sensor for quantitative normal stress measurements. The diffusion air bearing has a low inertia with high axial and radial mechanical stiffness.

The SPECTECH DSR was designed to be applications specific for testing of asphalt binders according to AASHTO TP5. The instrument features disposable plates and user-independent, automatic sample trimming.

## *Electronic unit*

The VISCOTECH DSR instrument electronics are contained within the mechanical unit, and the instrument is built around a dedicated, high-speed 32-bit CPU. This consolidation enhances performance and versatility due to having electrical connections on the motherboard bus rather than through cables to a separate electronics cabinet. In addition, valuable bench space is kept to a minimum. The motor control is based on digital rather than analog drive technology. The unit includes a built-in diagnostic system and quick diagnostic service port for service engineers. Also included is a modem port for remote-control operation and fault diagnostics for service. The electronics power supply is designed to operate on a line voltage of 180–260 V or 90–140 V and an operating frequency of 47–63 Hz.

## *Software package*

The standard software package is a true multitasking **Microsoft** (Redmond, WA) Windows™-based rheological software that has many advantages to the user. The computer is not dedicated to simply running the instrument and is available for other use when making measurements. It can be used for printing previous results, writing a report, or performing measurements with another instrument. The software runs under all Windows platforms.

The DSR software package offers measuring programs designed to be user friendly with few subdialogue levels and a general recognizable design. Preprogrammed test parameters and pass/fail test result limits are included. Additional experiment methods and analyses are available in the following test modes: viscometry, yield stress, constant rate, oscillation, oscillation stress sweep, oscillation strain control, creep/recovery, compare, analyze, time–temperature superposition, and spectrum transformation.

The software includes possibilities to link user-designed methods, including instrument setup and zero gapping using the project software. The dialogue windows have many storable, editable functions for unique testing requirements, and can be reset to default values using default buttons. An example is the Oscillation Frequency Step measuring program, where stresses, delay times, integration periods, and sample sizes can be set individually for all frequencies. Another example is the zooming function, which is presented in both Viscometry Stress Step and Oscillation Frequency Step, allowing any number of steps and increments to be selected. The instrument also performs controlled strain, and constant shear rate measurements, and includes automatic gap adjustments and compensation using the normal force sensor. This system enhances measurement reproducibility since the sample loading history is reproduced identically each time, using a constant loading force.

The VISCOTECH DSR is operated with a separate power supply unit that should be left on continu-

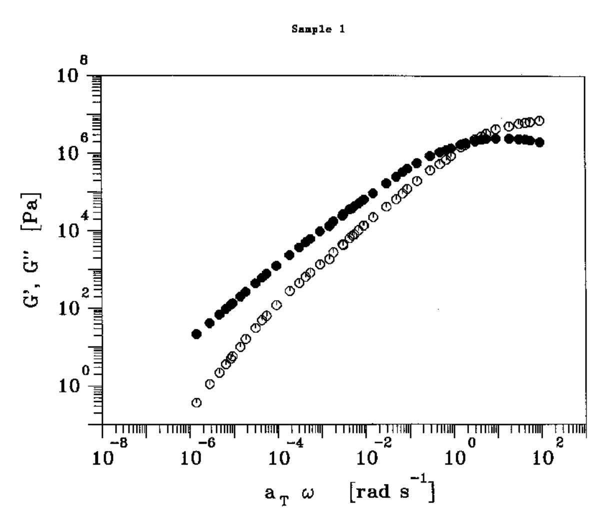


**Figure 9** *Master curve at 25 °C, sample 1.*

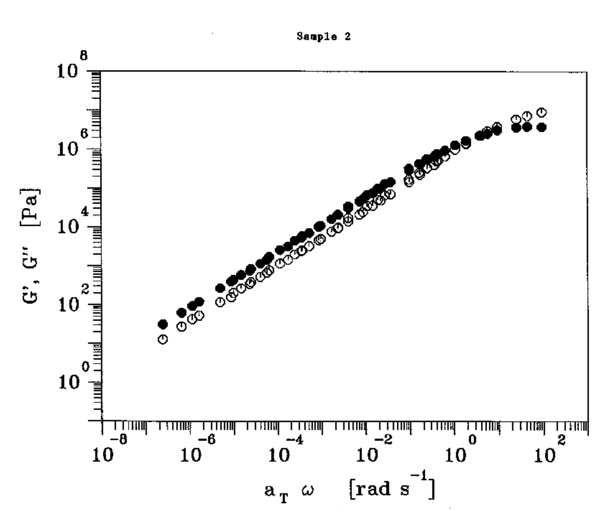


**Figure 10** *Master curve at 25 °C, sample 2.*

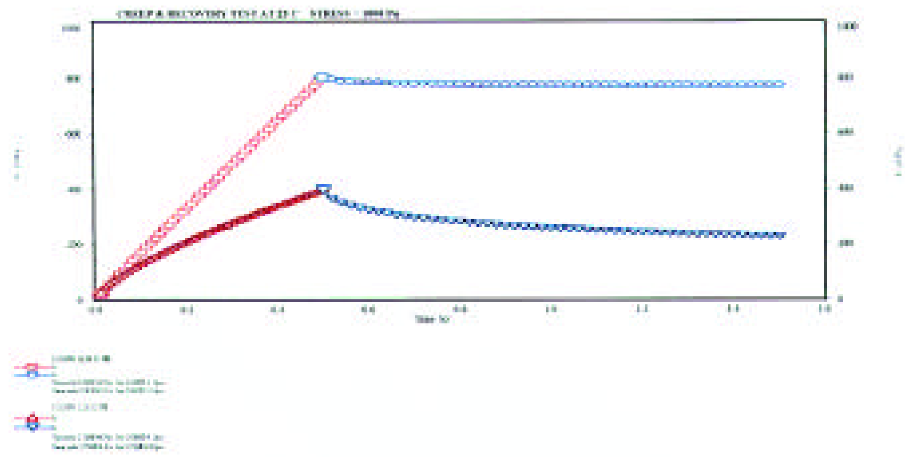

**Figure 11** *Creep/recovery results for samples 1 and 2.*

ously. This reduces startup times and makes it possible for the instrument processor to maintain values as gap and other user-defined settings.

The VISCOTECH DSR standard temperature control cell is a resistive heating and adiabatic cooling system. Other temperature cells are available including circulating fluid and cryogenic cooling, covering the range –180 to 500 °C. All measuring geometries are supported, i.e., cone/plate/parallel plate, concentric cylinder, and solid in torsion. A patented sealed cell for operating at elevated pressures with full oscillatory capabilities is available.

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*continued on p. 23*