TA Instruments

Rheometers

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TA Instruments, Worldwide

More worldwide customers choose TA Instruments than any competitor as their preferred rheometer supplier. We earn this distinction by best meeting customer needs and expectations for high technology products, quality manufacturing, timely deliveries, excellent training, and superior after-sales support.

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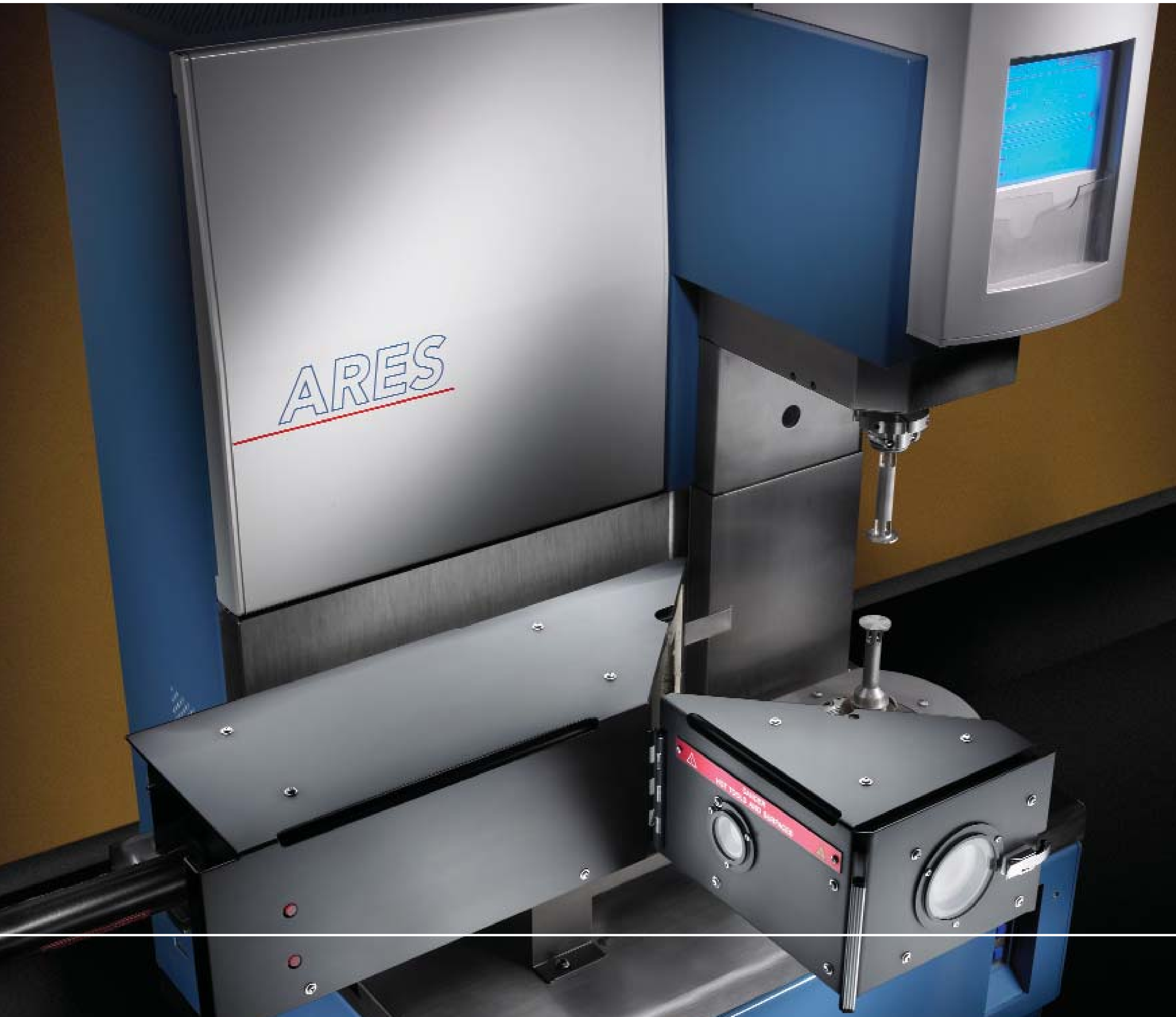
ARES

Pure · Robust · Industry Standard



ARES Rheometers

To the discerning rheologist, ARES is the brand of rheometer that offers the purest high performance rheological measurement due to its exclusive separate motor and patented transducer design, ease-of-use, and wide variety of advanced options. No other rheometer system employs a torque transducer to measure stress independently of the applied deformation. It is the only rheometer available that can offer viscosity independent, inertia-free oscillation measurements. With technology representing over 30 years of continual improvements based on input from the world's leading rheologists, ARES is the standard to which all other rheometers are held.

TA INNOVATIONS

First commercial rheometer for oscillation measurements

Torque and force rebalance transducer

Extensional viscosity fixture for shear rheometer platform

Introduction of multi-wave analysis

Tension control for enhanced performance in shear & torsion

First rheometer for testing fluids, melts & solids in shear & tension

Simultaneous rheology and optical analysis

Simultaneous rheology and dielectric analysis

Commercialized electrorheology

First to introduce multiple extension test

Arbitrary Waveform

ARES-LS

The ARES-LS is our most advanced rheometer, featuring our High Performance LS air bearing motor and patented Force Rebalance Transducer™ (FRT). The ARES-LS1 includes the 1KFRTN1, with a dynamic range of six decades. It is the only rheometer in the world capable of providing inertia-free dynamic measurements of low viscosity structured fluids over a wide frequency range, independent of the material's viscosity. The ARES-LS2 with the 2KFRTN1 is an outstanding choice for medium to high viscosity materials including polymer melts and solids.

	ARES-LS2	ARES-LS1
Motor	High Performance LS	High Performance LS
Angular Velocity Range	2×10^{-6} - 200 rad/s	2×10^{-6} - 200 rad/s
Strain Amplitude	5 µrad - 500 mrad	5 µrad - 500 mrad
Angular Frequency	10^{-5} - 500 rad/s	10^{-5} - 200 rad/s
Transducer	2KFRTN1	1KFRTN1
Torque Range	2 µN.m - 200 mN.m	0.2 µN.m - 100 mN.m
Normal/Axial Force Range	0.002 - 20 N	0.002 - 20 N
Auto Gap Set	Standard	Standard
Motor Bearings	Jeweled Air	Jeweled Air
Temperature Control Options		
Peltier Plate	-40 to 150 °C	-40 to 150 °C
Bath	-10 to 150 °C	-10 to 150 °C
Forced Convection Oven (FCO)	-150 to 600 °C	-150 to 250 °C

ARES

The ARES is an extremely capable and versatile all-purpose rheometer that incorporates the Standard HR Motor and the 2KFRTN1 Force Rebalance Transducer™. The ARES is appropriate for characterizing a diverse variety of materials including polymer melts, solids and reactive materials, as well as a broad spectrum of medium to high viscosity fluids. Measurements can be made over a wide range of temperatures using any of the available temperature control options.

	ARES
Motor	Standard HR
Angular Velocity Range	10^{-3} - 100 rad/s
Strain Amplitude	5 µrad - 500 mrad
Angular Frequency	10^{-5} - 500 rad/s
Transducer	2KFRTN1
Torque Range	2 µN.m - 200 mN.m
Normal/Axial Force Range	.002 - 20 N
Auto Gap Set	Standard
Motor Bearings	Mechanical
Temperature Control Options	
Peltier Plate	-40 to 150 °C
Bath	-10 to 150 °C
Forced Convection Oven (FCO)	-150 to 600 °C

ARES-RFS

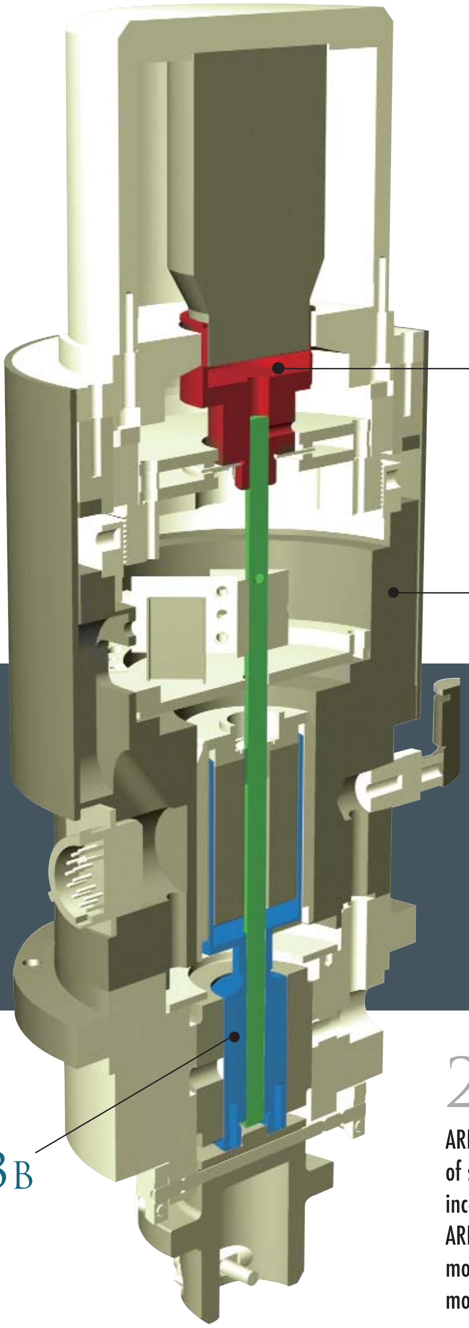
For characterization of both the structure and flow properties of simple and complex fluids, the ARES-RFS is an excellent choice. Configured with the 1KFRTN1 Force Rebalance Transducer™ and the Standard HR motor, the ARES-RFS can solve the most demanding fluid rheology problems including dynamic oscillatory and shear rate controlled measurements on low viscosity structured fluids. The ARES-RFS is normally configured with the Peltier or Recirculating Bath temperature control option.

ARES-RDA

The ARES-RDA is a robust rheometer designed specifically for general rheological characterization of polymers, including thermoplastics, thermosets, and elastomers. The 2KSTD spring transducer is a durable design with excellent sensitivity, linearity, and stiffness over a wide dynamic range. Normally configured with the Forced Convection Oven (FCO), the ARES-RDA is the ideal choice for many analytical and QC labs with polymer characterization needs.

	ARES-RFS	ARES-RDA
Motor	Standard HR	Standard HR
Angular Velocity Range	10^{-3} - 100 rad/s	10^{-3} - 100 rad/s
Strain Amplitude	5 µrad - 500 mrad	5 µrad - 500 mrad
Angular Frequency	10^{-5} - 200 rad/s	10^{-5} - 500 rad/s
Transducer	1KFRTN1	2KSTD Spring
Torque Range	0.2 µN.m - 100 mN.m	20 µN.m - 200 mN.m
Normal/Axial Force Range	.002 - 20 N	Qualitative
Auto Gap Set	Standard	Standard
Motor Bearings	Mechanical	Mechanical
Temperature Control Options		
Peltier Plate	-40 to 150 °C	-40 to 150 °C
Bath	-10 to 150 °C	-10 to 150 °C
Forced Convection Oven (FCO)	-150 to 250 °C	-150 to 600 °C

ARES Technology



1 Torque Transducer

ARES rheometers are unique in that they are the only commercial rheometers available which use separate torque transducers to make stress measurements independent of the applied stress or strain rate. By separating the transducer, the motor inertia is decoupled from the torque measurement permitting accurate and precise viscosity-independent oscillatory measurements. The ARES-RDA is configured with a robust cost-effective spring transducer, which provides outstanding performance for general rheological testing of polymers. All other ARES rheometers are configured with one of two patented Force Rebalance Transducers™ (FRT). In an FRT, a capacitive position sensor detects angular movement and a rotary motor measures the reaction torque to drive the geometry back to the original position. The FRT provides a wide torque range (up to 6 decades) with high torque sensitivity.

ARES

The ARES series represents a family of unique separate motor and transducer rheometers offered exclusively from TA Instruments.

2 Drive Motor

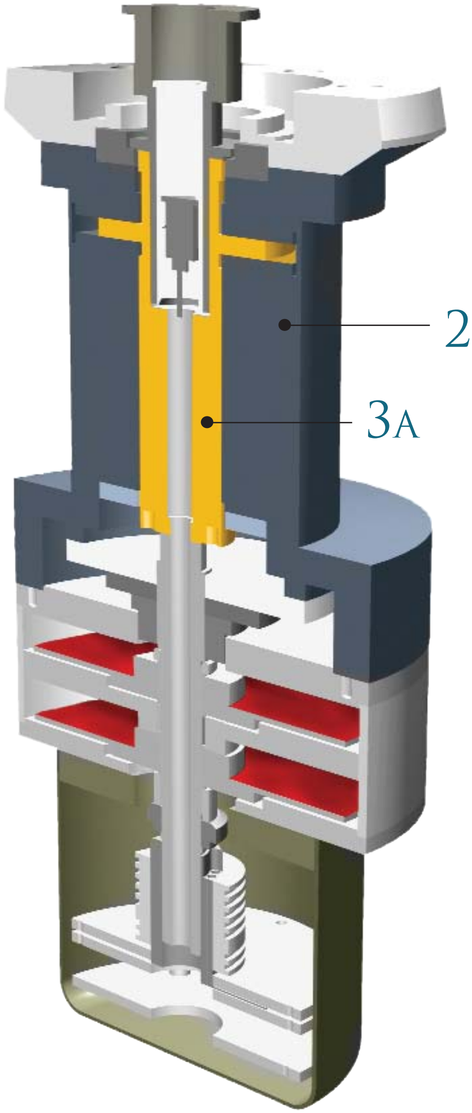
ARES rheometers incorporate one of two direct-drive, high torque motors for control of strain, strain rate, and oscillation frequency. The ARES, ARES-RDA, and ARES-RFS incorporate a high resolution (HR), motor with a broad performance range. The ARES-LS models are equipped with the high performance low shear (LS) air-bearing motor with inductosyn drive technology, which provides for unmatched low speed motor control.

3 Air Bearings

The high performance ARES-LS motor incorporates sapphire "jeweled" air bearings [3A]. These air bearings provide a very stiff, yet low friction means of supporting rotational motion. The stiff, smooth motion provided by the air bearing is necessary for accurate transient normal force measurements. ARES FRT transducer motors are supported with fluted bearings [3B]. These bearings provide the low friction and high stiffness in a compact design required for the small rotary motor.

4 Normal Force Transducer

Quantitative normal force is measured on all ARES rheometers with patented force rebalance transducers. Normal force may be generated when shearing a viscoelastic sample, which acts to push the rheometer plates apart. A capacitive position sensor detects this linear motion and a motor provides a reaction force to rebalance, or drive, the geometry back to the null position. This measurement technique, coupled with the only rheometer on a steel frame, provides for the most accurate normal force measurements available. Where experimentally necessary, such as during sample loading or for compensation of volume change during testing, normal force can also be controlled with great precision.



•Frame Assembly and Linear slide

ARES rheometers are built for maximum stiffness to ensure all deformation is applied to the sample. The ARES rheometer is the only commercial rheometer that houses components in a rigid, cast steel frame. The transducer (upper head) is attached to the steel frame via a precision linear slide. The slide is driven by a motor with a preloaded spindle to prevent backlash. An optical encoder measures the position of the movement of the linear slide, precisely positioning attached geometries to an accuracy of 1 micron.

ARES Temperature Control Options

Peltier Plate

The Peltier Plate temperature control option is available for all models of the ARES Rheometers, and can be used with parallel plate and cone and plate geometries. With a temperature range of -40 to 150 °C, it is the most common temperature control option for a variety of fluid applications. The open design facilitates easy sample loading and cleaning of geometries. A PRT (platinum resistance thermometer) sensor is positioned in the middle of the lower sample plate and ensures accurate measurement and control of sample temperature. Maximum heating rate is 30 °C/min with a temperature accuracy of +/- 0.1 °C.

Recirculating Fluid Bath

The Recirculating Fluid Bath option can be used with parallel plate, cone and plate, and concentric cylinder geometries. Concentric cylinders are especially useful for very low viscosity fluids, dispersions of limited stability and for applications where fluid/solvent evaporation may be a concern. This option is available for all models of the ARES and can be supplied with a computer-controlled circulator for automated operation. The temperature range is -10 to 150 °C with the appropriate circulating fluid.

Forced Convection Oven (FCO)

The Forced Convection Oven (FCO) is an air convection oven with dual-element heaters and counter-rotating airflow for optimum temperature stability, and is used primarily for polymer melts and solids. The temperature range is -150 to 600 °C with heating rates up to 60 °C/min. An optional liquid nitrogen cooling device is required to achieve subambient operation to -150 °C. The FCO can be used with parallel plate, cone and plate, or torsional clamps for solids, and the new Extensional Viscosity Fixture (EVF). Disposable plates are available for thermoset cures. The FCO includes a sight glass so that the sample can be viewed during the experiment.

ARES

ARES Accessories

Extensional Viscosity Fixture, (EVF)

The EVF is a patent pending extensional viscosity fixture for measurements of the elongation viscosity of high viscosity materials, such as polymer melts, dough, adhesives, etc. The EVF can be used on ARES systems configured with the convection oven and provides best performance with an FRT transducers. The fixture consists of a fixed and rotating drum, which winds up the sample at constant strain rate, while measuring the force generated in the sample. Since the torque measurement is decoupled from the motor, no bearing friction correction is required. The maximum Hencky strain with one rotation is four, and the maximum operating temperature is 350 °C.

Optical Analysis Module OAM II

The OAM II offers both parallel plates and concentric cylinder flow cells for fluids and melts. An optical train directs light through the flow cell at normal incidence. Transmitted light is received directly by a detector for dichroism measurements, or after passing through a circular polarizer for birefringence measurements. Applications include determination of the stress optical coefficient, separating the component dynamics in compatible polymer blends, analysis of polydomain structure in liquid crystals, and reorientation of internal structure under flow in dispersions (emulsions, suspensions).

Waveform and Fast Sampling

ARES rheometers can be equipped with a data monitor and fast sampling option. During oscillation and steady shear testing, the torque, strain, and normal force raw signals are monitored. The benefit is the detection of non-linear sample behavior and determination of steady state in real-time, allowing the operator to make intelligent decisions about test parameter selection and accurate data interpretation. The fast data acquisition option allows for data collection rates up to 500 data points per second in oscillation experiments. This is ideal for tracking rapidly changing systems, such as UV curable adhesives and coatings which may react in less than one second.

UV-Curing and Photocuring Accessory

The Photocuring Accessory includes upper tool with reflecting mirror and quartz plate, lower plate, light source mount and collimator, 5mm waveguide and remote radiometer/dosimeter. A high-pressure mercury lamp UV light source is included with the UV-curing accessory. Both accessories can be used with the ARES Peltier and Bath temperature control systems. Disposable plates are available for hard UV coatings that cannot be removed from the quartz plates once cured. For extremely fast reacting UV materials, the fast sampling option allows for data collection rates of up to 500 data points per second.

Dielectric Analysis Option, (DETA)

The DETA provides simultaneous dielectric (permittivity, loss factor, tan delta) and rheological data (G′, G″, tan delta) during time and temperature-dependent testing. This option is available with or without the Agilent 4284A LCR bridge, with frequency range of 20 Hz to 1 MHz and voltage range of 0.01 to 10 volts. Up to 10 frequencies can be entered for a test. Electrodes are 25 or 40 mm diameter stainless steel plates. The DETA option will work with the FCO up to temperatures of 350 °C.

Electro-Rheological (ER) Analysis Option

The ER option allows the study of rheological properties of materials under the influence of AC/DC voltages. The ER option is designed for use under ambient conditions or with the Peltier environmental control system, and is now available for use with both 1KFRT and 2KFRT transducers. The option includes upper tool insulator assembly, oscilloscope, safety panels, interlocks, a high voltage/current amplifier with ability to apply up to 4000 Volt potential, up to 15 mA current (both AC & DC) and slew rate of 100 V/microseconds, and gain bandwidth of 35 MHz.

Applications

Flow Curve for Dispersions

A generalized flow curve for dispersions is illustrated in Figure 1. TA rheometers generate flow curves by applying a stress ramp (or shear rate) and measuring the shear rate (or stress). Flow curves can also be produced using "steady state" flow where each viscosity data point is generated at a constant stress after equilibration. The data generated provides information on yield stress, viscosity, shear thinning, shear thickening, thixotropy, and correlates to processing and product performance. Simple techniques like spindle viscometers can only measure a point or a small part of the total flow curve.

Flow Curve for Polymers

Figure 2 shows a generalized flow curve for polymers and corresponding process shear rate ranges. A polymer's molecular weight greatly influences its zero shear viscosity, while its molecular weight distribution and degree of branching affect its shear rate dependence. These differences are most apparent at low shear rates not possible with melt flow index or capillary devices. TA rheometers can determine molecular weight based on the measured zero shear viscosity. Cox-Merz and TTS can be used to extend the data to higher shear rates.

FIGURE 1

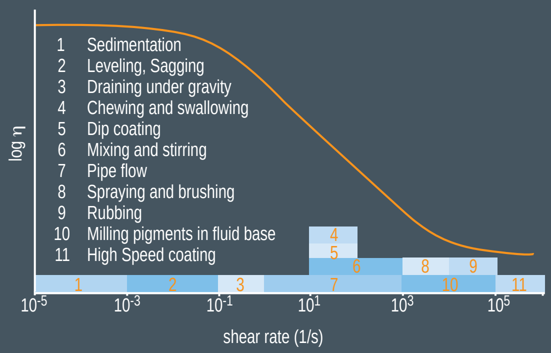
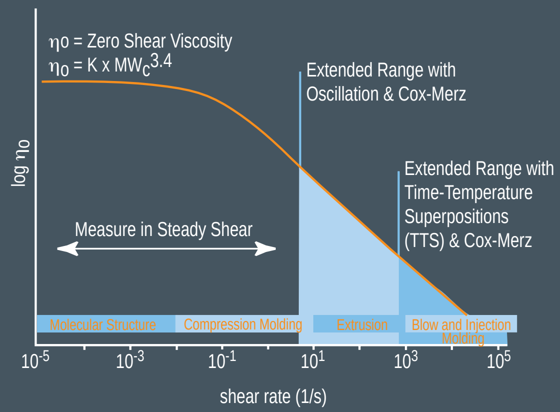


FIGURE 2



Viscoelastic Properties

The viscoelastic properties of polymer melts are commonly studied in the dynamic oscillation mode. Figure 3 illustrates a viscoelastic fingerprint for a linear homopolymer and shows the variation of the storage modulus (G') and loss modulus (G'') with frequency. Since polymer melts are viscoelastic, the mechanical response will be time dependent, so low frequencies correspond to long times. TTS is used to extend the range of data to higher and lower frequencies and to build a master curve at a reference temperature. The magnitude and shape of the G' and G'' curves depends on the molecular structure of the polymer.

Oscillation Stress or Strain Sweeps

Key viscoelastic parameters (G', G'', η_*, tan delta, etc.) can be measured in oscillation as a function of stress, strain, frequency, temperature or time. Figure 4 illustrates a common oscillation stress sweep used to determine the linear viscoelastic region (LVR) and to investigate dispersion stability. In the LVR, the material responds linearly to the stress or strain (elastic) and the structure remains unchanged. The rapid drop of the modulus with stress represents breakdown of the material's structure. Once the structure has been broken, G' becomes dependent on the stress. Similar studies can be conducted as a function of strain.

FIGURE 3

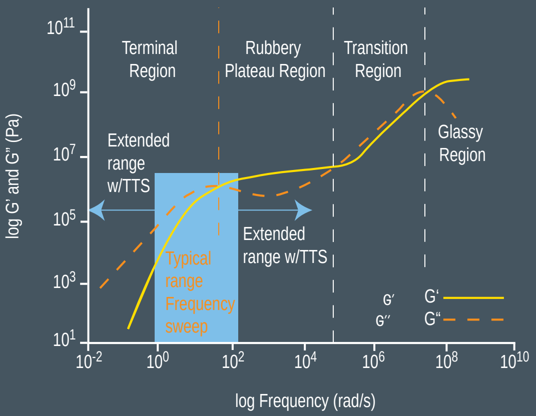
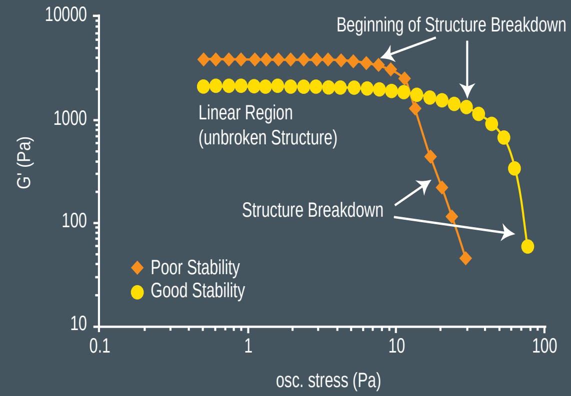


FIGURE 4



Applications

Dynamic Mechanical Properties of Solids in Torsion

The ability to characterize the viscoelastic properties of solids in torsion is a feature of TA Instruments' rheometers, as illustrated in Figure 5 for polycarbonate (PC). Transitions or relaxations of molecular segments are observed as step changes in the storage modulus, and as peaks in the loss modulus and damping. The magnitude and shape of the storage modulus (G'), loss modulus (G'') and damping (tan delta) will depend on chemical composition, crystallinity, molecular structure, degree of cross-linking, and the type and amount of fillers.

Transient Tests (Creep and Stress Relaxation)

In a creep recovery test, illustrated in Figure 6, a constant stress is applied to the sample and the resulting strain is measured over time. The stress is then removed and the recovery (recoil) strain is measured. For polymer melts, the zero shear viscosity (η_0) and equilibrium recoverable compliance (J_{eo}) can be determined. Creep is a sensitive technique and best suited for the unmatched stress control performance of the AR. In a stress relaxation test, a strain is applied and stress is measured as a function of time yielding stress relaxation modulus G(t). Stress relaxation can be performed on all ARES and on the AR-G2 and AR 2000ex with direct strain control.

FIGURE 5

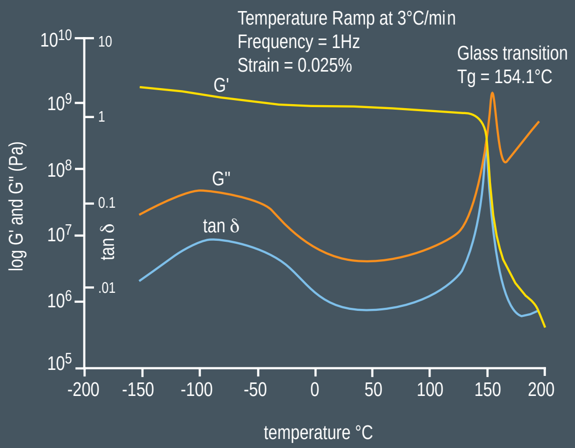
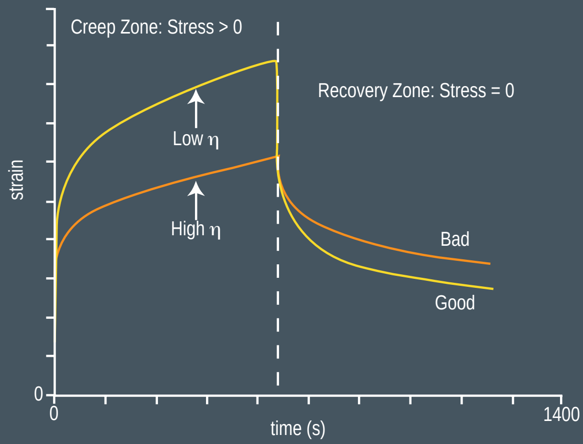


FIGURE 6



Dynamic Oscillation on Low Viscosity Fluids Using ARES

An advantage of the ARES design is that the motor generates the torque to overcome the viscosity of the material, as well as the inertia of the sample holder. As a result, the ARES can be used to conduct inertia free measurements on the viscoelastic properties of very low viscosity fluids. Figure 7 shows an example of this for a polymer solution, where the viscoelastic parameters are determined using a frequency sweep up to 100 rad/s with no inertial effects.

AR-G2 Nano-Torque Measurements in Stress & Strain Control Oscillation

The minimum torque on rheometer designs, which use only air bearings, is specified at a higher value for controlled stress (CS), as compared to controlled rate or strain (CR). The new magnetic thrust bearing and advanced drag cup technologies incorporated on the new AR-G2 allow for ultra-low, nano-torque control in both controlled stress and controlled strain modes. This unprecedented torque performance is shown in Figure 8. Note, viscosity and displacement values at instrument torque levels of 3 nN.m.

FIGURE 7

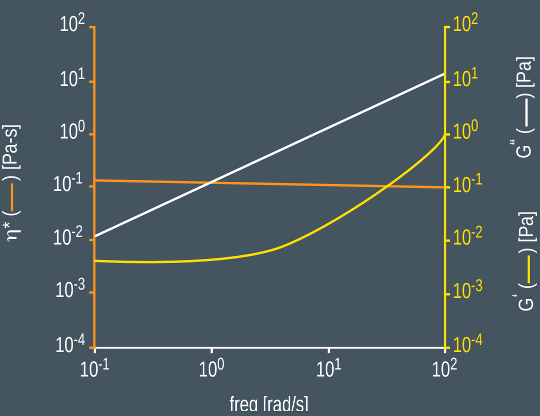
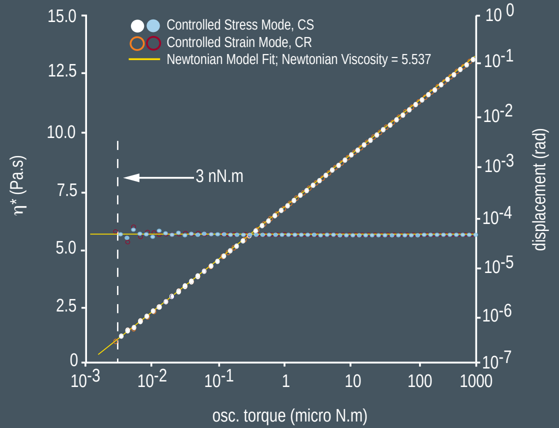


FIGURE 8



Applications

Extensional Viscosity Measurements on ARES

Extensional viscosity is fundamentally important in many polymer-processing techniques such as blow-molding, fiber spinning, and injection molding. The EVF is a polymer melt elongation fixture that transforms an ARES oven system into a shear and extensional rheometer. The EVF uses a unique patented dual cylinder, or drum, wind-up technique. Figure 9 shows the data on a LDPE sample, superposed with three times the shear and complex viscosity measured at a rate of 0.01 1/s and frequency w = 1/t. The EVF clearly shows excellent data over a wide range of rates.

Monitoring UV Curing Systems

UV curing materials react within seconds while the modulus increases several decades. Using a special UV testing option and fast acquisition of the oscillation data, G′, G″ or η^*, TA rheometers can monitor the modulus change during these reactions. Special test geometries are used to distribute the UV light homogeneously onto the sample. The dynamic data points are acquired as fast as every 2 ms. Figure 10 illustrates the cure of an acrylate based PSA, monitored as a function of the UV intensity over time.

FIGURE 9

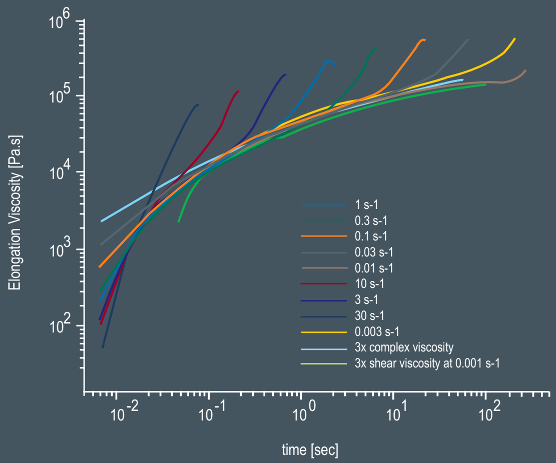
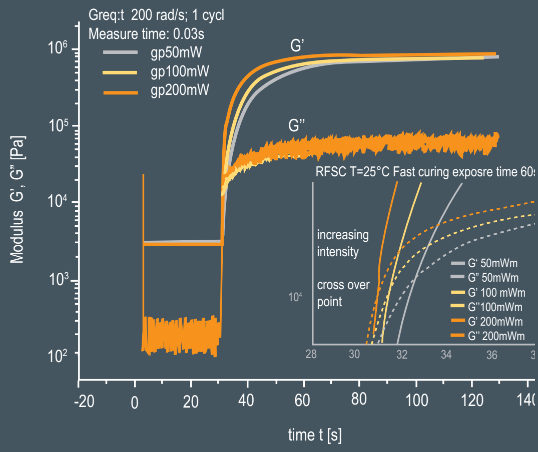


FIGURE 10



Transient Viscosity and 1st Normal Stress Coefficient in a Step Rate Experiment

One of the most challenging rheological measurements to be made on a rheometer is the transient viscosity and 1st normal stress coefficient of polymer melts. In order to eliminate secondary flows, which primarily affect the normal force, the axial compliance of the instrument setup must be extremely low. Such challenging measurements are easily obtained using an ARES with patented rebalance transducer with a maximum compliance of 0.1μm/N. Figure 11 shows results of a series of step rate experiments performed over a shear rate range from 0.1 to 10 s/1. Both, transient viscosity and 1st normal stress coefficient at all rates superpose very well at short times. At longer times, the viscosity and normal stress coefficient traces diverge due to the nonlinear material response. A stress overshoot is observed in the transient viscosity at 5 and 10 s-1 for this LDPE melt.

Creep and Recovery Measurements of Polymer Melts

Creep and Recovery measurements are extremely sensitive for long time relaxation processes in polymers. During these experiments, a constant stress is applied to the sample and the resulting strain is measured with time. The stress is then removed and the recovery (recoil) strain is measured. The zero shear viscosity (n_0) for polymer melts can be determined from the total non-recoverable and the equilibrium compliance (Je_0) from the recoverable strain. Only the AR-G2 provides extremely low residual torques and creep braking option both of which are essential for accurate measurements of small elastic strain contributions. Figure 12 shows the compliance (measured strain/applied stress) and the recoverable compliance for narrowly distributed polystyrene. Extremely accurate measurements are easily obtained on this system over a stress range of 10 to 10,000 Pa. The result is independent of stress, i.e. this polymer melt exhibits linear viscoelastic behavior.

FIGURE 11

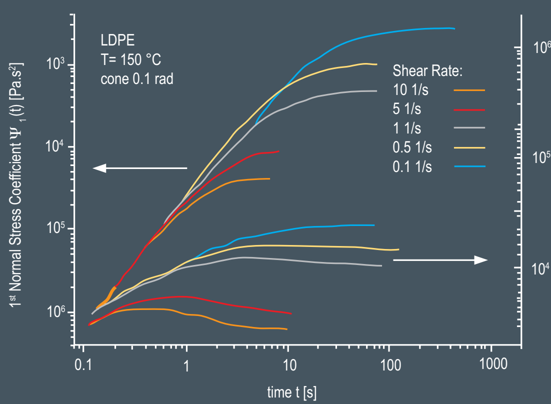
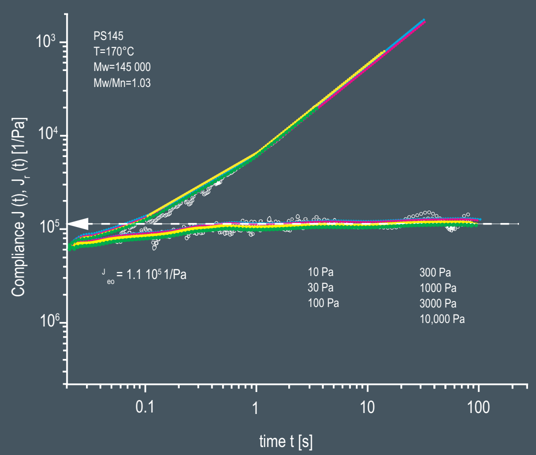


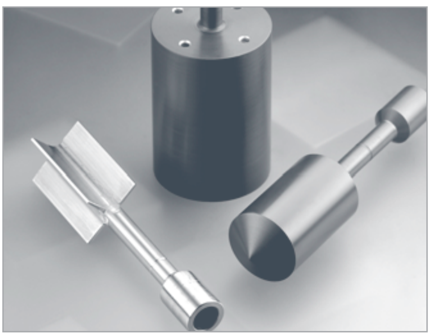
FIGURE 12



Geometries

TA Instruments offers a wide range of measurement geometries including parallel plate, cone and plate, concentric cylinder, disposable, and torsion solid clamps.

Parallel plate and cone and plate geometries are available for both the ARES and AR Rheometers in an extensive variety of diameters and cone angles. Materials of construction include stainless steel, aluminum, plastic (Acrylic/PPS), or titanium. Disposable plates and cones are also available for applications such as thermoset curing.



A wide selection of concentric cylinder geometries are available for both the ARES and AR Rheometers. Options include conical DIN, recessed, vaned, and double wall. For the ARES rheometers, the geometries are constructed of stainless steel and titanium. For the AR rheometers, stainless steel and anodized aluminum are used.

A variety of other geometries are available for both the ARES and AR Rheometers, including clamps to measure solids in torsion using the high temperature ovens and immersion clamps. The ARES is available with many specialty fixtures including linear tack-testing fixtures, glass plates for optical measurements, and film/fiber fixtures.

Notes

Technical Support

Customers prefer TA Instruments because of our reputation for after-sales support. Our worldwide technical support staff is the largest and most experienced in the industry. They are accessible daily by telephone, email, or via our website. Multiple training opportunities are available including on-site training, seminars in our application labs around the world and convenient web-based courses.

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