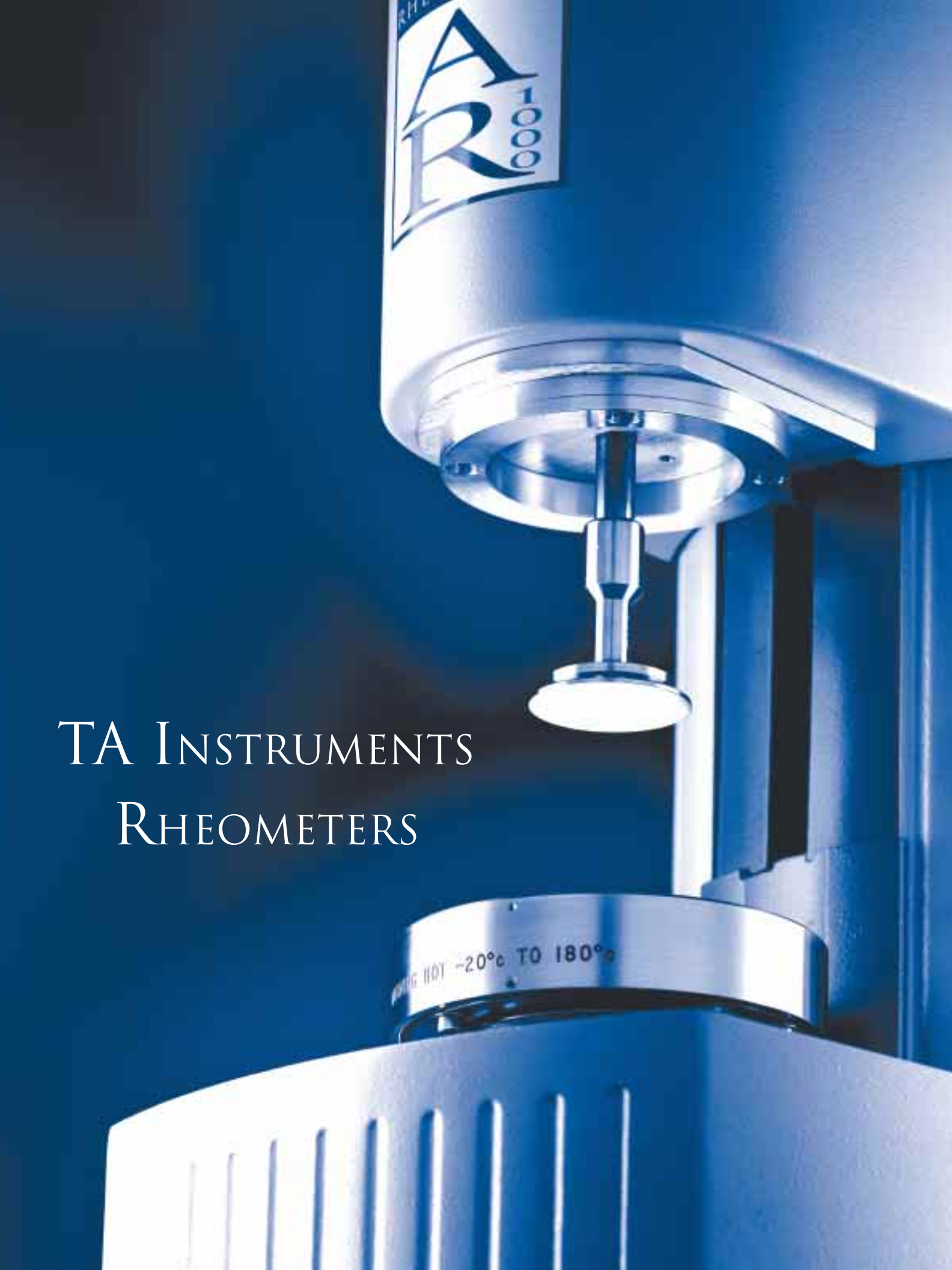


TA Instruments
Rheometers

The TA Instruments Rheometer

Sensitive, Accurate, Rugged, and Reliable, these words describe a TA Instruments rheometer. The AR 1000 and AR 500 are fourth generation products from the pioneer of controlled stress rheology. Designed with the customer in mind, and backed by the superior customer support which is the hallmark of TA Instruments, these rheometers have set a new standard for performance.

The AR 1000 is TA Instruments top of the line research grade rheometer. The unique motor design and advanced material air bearing provide superior torque performance. The AR 1000 can be equipped with multiple temperature control options, normal force sensor, and custom geometries. It is well equipped to handle the most demanding rheological applications.

The AR 500 is a general purpose rheometer that outperforms other vendors' research grade systems. The AR 500 is an upgradeable system that can grow as your applications expand. It is the ideal rheology system for users interested in a robust, cost-effective system with outstanding basic performance.

Are you ready to transfer rheological tests from the lab to manufacturing facilities, or quality control labs? The QCR II is a robust rheometer that easily automates the analysis of a broad range of samples.

AR 1000 & 500 Technical Specifications

	AR 500	AR 1000
Minimum Torque	1.0 µNm	0.1 µNm
Maximum Torque	50 mNm	100 mNm
Frequency Range	0.0001 to 40 Hz	0.0001 to 100 Hz
Minimum Angular Velocity	10^{-8} radians / second	10^{-8} radians / second
Maximum Angular Velocity	10^{2} radians / second	10^{2} radians / second
Displacement Resolution	0.62 µ radians	0.62 µ radians
Maximum Angular Deflection	± 1300 radians	± 1300 radians
Air Bearing	Jet	Porous Carbon
Auto Gap Set	Standard	Standard
Gap Resolution	0.06 µm	0.06 µm
Normal Force Range	0.01 to 50 N	0.01 to 50 N
	Temperature Control Options	
Environmental Test Chamber	-150 to 400°C	-150 to 400°C
Extended Temperature Module	-100 to 400°C	-100 to 400°C
Standard Peltier	-10 to 99.9°C	-10 to 99.9°C
Extended Peltier	-20 to 180°C*	-20 to 180°C*
Fluid Jacket with Circulator	-20 to 150°C	-20 to 150°C

*Lower temperature can be reduced to -40°C by use of a suitable fluid in the external circulator.

Instrument Design Features and Benefits

– A Rheometer is only as good as its ability to apply torque and measure displacement. Our engineers use custom designed components and proprietary materials (described below) to achieve unequalled stress and strain performance.

•***Drag Cup Motor*** Non-contact, with an excellent torque to inertia ratio, and no overheating problems, the drag cup motor provides torque over a very wide range. **Benefits:** A wide variety of materials can be studied from very low viscosity materials to polymer melts and solids.

•***Air Bearing*** TA Instruments' unique, custom-designed, air bearings provide frictionless support for the drive shaft and measuring geometry. Our long-life jet bearings (AR 500) and porous carbon bearings (AR 1000) provide low levels of residual torque, and ultra low inertia. *Rotational Mapping* automatically corrects for residual torque in the system. **Benefits:** TA bearings provide excellent torque resolution, and allow the application of a wide range of torque (stress). The porous carbon air bearing on the AR 1000 extends the low torque performance of the instrument.

•***Optical Encoder*** Measures angular deflection with high resolution. **Benefit:** Sample measurements can be conducted at low shear rates, small displacements (strain), and high velocities.

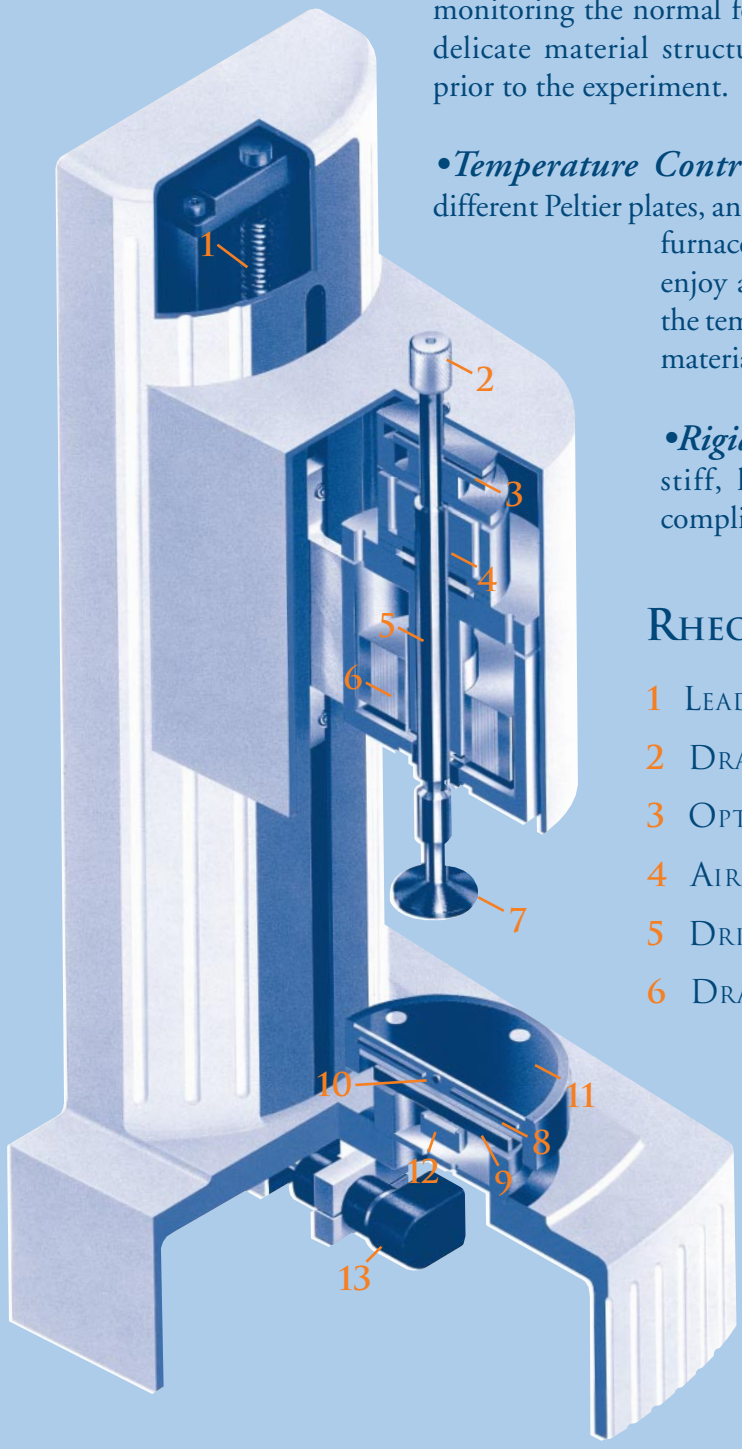
•***Normal Force Transducer*** This highly sensitive, ultra stiff transducer located below the sample plate measures a wide range of normal force exerted by a sample, without a change in gap. **Benefits:** Quantitative normal forces exhibited by materials with different viscoelastic properties are measured. Normal forces generated during sample loading can be monitored.

•***Linear Ball Slide*** Mounts the motor and air bearing to the casting. The high precision slide is driven vertically by a motor in the base. A second optical encoder is located in the base to measure the movement of the slide. **Benefits:** Precise geometry location relative to the sample is assured. The long travel permitted by the ball slide allows for a large working space to simplify sample loading and cleaning.

•***Auto Gap Set*** The software provides automatic setting of gap, and programmed gap closure via several methods (linear, exponential). *Thermal Gap Compensation* automatically corrects for any change in sample gap due to thermal expansion. **Benefits:** Automatic and reproducible setting of the sample gap ensures accuracy and reproducibility. By monitoring the normal force exerted by the sample during closure, delicate material structures are protected rather than destroyed prior to the experiment.

•***Temperature Control*** Interchangeable options include two different Peltier plates, an induction heating system (ETM), a radiant furnace (ETC), and a fluid jacket. **Benefits:** Users enjoy a wide temperature range, and can choose the temperature control option that best suits their materials.

•***Rigid one-piece aluminum casting.*** This stiff, high mass casting ensures low system compliance.



Rheometer Schematic

1 Lead Screw
2 Draw Rod
3 Optical Encoder
4 Air Bearing
5 Drive Shaft
6 Drag Cup Motor
7 Measuring Geometry
8 Peltier Elements
9 Heat Exchanger
10 Pt 100 Temperature Sensor
11 Durable Chromium Surface
12 Normal Force Tranducer
13 Auto Gap Set Motor and Encoder

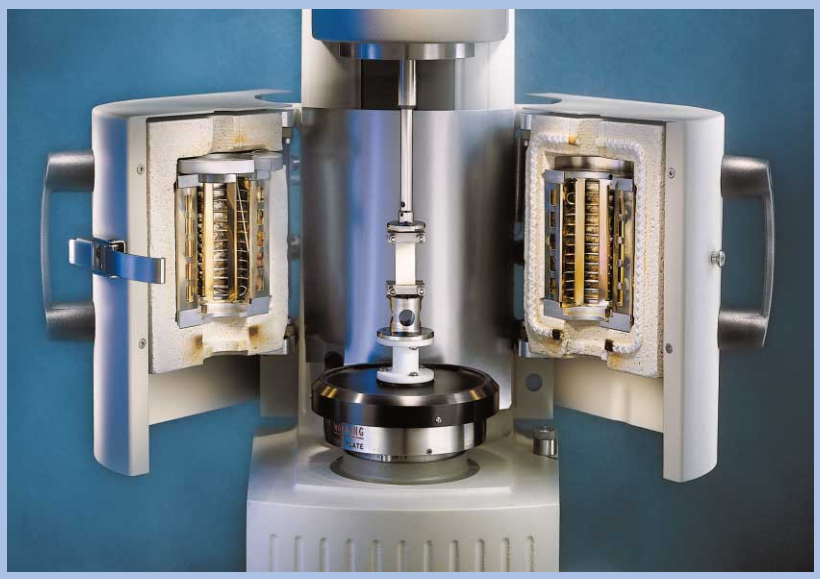
AR 1000 & 500 Temperature Control Options

Peltier Plate

The Peltier Plate is the standard temperature control device for the TA Instruments' Rheometers and is available in two temperature ranges. The Standard Peltier Plate has a temperature range of -10 to +99.9°C while the Extended Peltier Plate operates from -20°C to +180°C. Over these ranges they will provide an accuracy of ± 0.1°C and a typical heating rate of 20°C per minute. The Peltier Plate is the rheologist's choice for most fluid applications. Ideally configured for parallel plate or cone and plate systems, the use of narrow gaps permits the rapid conduction of heat through the sample. In addition, the open design of the Peltier Plate facilitates easy sample loading and cleaning of the durable chromium surface. A Pt100 sensor is positioned at the center of the sample plate to ensure accurate measurement and control of sample temperature.

Concentric Cylinders

The Fluid Jacket Temperature System for the AR 1000/500 Rheometers is designed to provide precise temperature control for concentric and double concentric cylinder geometries over a wide range of temperatures. Generally, cone & plate or parallel plate geometries are the first choice for most rheological measurements because of their small sample volume, rapid temperature equilibration, and easy cleanup. Concentric cylinders are best used for low viscosity samples, and those with large particles and/or limited stability. A range of systems (conical, recessed, vaned, and double concentric) are available for optimum performance. The system is self-aligning, and is quick and easy to change from cone & plate to concentric cylinders use.



Environmental Test Chamber

The ETC provides controlled radiant heating and cooling (LN_2) over the temperature range -150 to 400°C with heating rates up to 15°C/min (depending on conditions). Its main use is in the analysis of polymer melts using cone and plate and parallel plate geometries. Disposable plates are available for thermoset studies. The ETC is commonly used to analyze solids in torsion and can accommodate standard sample sizes specified in ASTM D4065. It thus allows the rheologist to measure diverse samples over their complete viscoelastic spectrum.

Extended Temperature Module

The ETM is designed to provide rapid heating and cooling (up to 120°C/min) with very precise temperature control over a wide temperature range (-100 to 400°C). This outstanding performance is possible because the ETM is a unique induction heating system. It is an ideal system for analyzing the isothermal cure of thermosetting polymers where it is critical to rapidly reach and stabilize at the test temperature. Additionally, many applications require fast heating rates to simulate process conditions. The ETM is ideally suited for these applications, as well as for any laboratory requiring high sample throughput.

Applications

The AR 1000 and AR 500 Rheometers with powerful, user-friendly Rheology Advantage 32-bit software and appropriate accessories combine to provide rapid characterization (with complete mathematical data modeling) of a broad range of materials from water to asphalt (~12 decades in viscosity). Commonly analyzed groups of materials include oils, gels, dispersions, pastes, slurries and polymers (melts and solids). Rheology is used in new product research, prediction of end use properties, competitive comparisons, selection of processing conditions, and quality control. The major areas of interest today, the technical challenges faced, and related rheological solutions using TA Instruments rheometers are shown. The analytical methods used to determine the shown rheological properties are commonly performed in Flow (Steady State Flow or Continuous Ramp), Creep and Oscillation modes. More sophisticated modes and data analysis techniques are also available (e.g. multiwave and time-temperature superposition). Illustrative examples of the common modes of operation using data from dispersions and polymers samples are also presented. The choice between the AR 1000 and AR 500 for a particular test will largely depend upon the performance required and the viscosity of the sample. For advice on an optimum configuration contact your local TA Instruments representative. More information can be obtained from our applications literature available on CD-ROM or directly from our website at http://www.tainst.com.

Major Applications
Polymers - Thermoplastics
Polymers - Thermosets
Polymers - Elastomers
Adhesives
Coatings - Paints
Coatings - Inks
Coatings - Powders
Foods - Pastes, Gels, Pastes, Gels, Dispersions (Suspensions, Emulsion)
Pharmaceuticals & Personal Care products - Pastes, Gels Dispersions (Suspensions, Emulsion)
Ceramics - Slurries
Oils, Greases, Lubricants

Process/Product Concerns	Related & Measurable Rheological Properties
Processability and Product Performance	Viscosity, Shear Thinning, Elasticity, Compliance
Die Swell	Normal Force
Structure (MW, MWD)	Elasticity, Viscosity Profile, Zero Shear Viscosity (η_0)
Effects of Branching, Fillers, Melt Flow	Changes in η_0, Elasticity, Compliance
Regrind Materials - Detection & Use	Comparison of Viscoelastic Properties (η_0, G', G", tan δ, compliance) with virgin material
Minimum Viscosity	Minimum in Viscosity Profile
Gel Point (Time / Temperature)	Intersection of Storage (G') & Loss (G") Moduli
Cure Profile / Cure Kinetics	Examine Modulus Profile with Temperature or Time (G', G" vs T or t)
Cross-link Density	Examine Plateau Modulus (G')
Cross-link Density	Examine Plateau Modulus (G') or Complex Viscosity (η^*) Profiles
Effect of Fillers	Comparison of Viscoelastic Properties (G', G", η^*)
Effect of Compounding	Strain Dependence of the Material (Length of Linear Viscoelastic Region)
Tire Traction / Tire Wear	Examine G', G", tan δ
Tack and Peel Characteristics	Frequency / Time Dependence of G', G"
Dalquist Criterion (pressure sensitive)	Examine Plateau Modulus - is G' in acceptable range?
Ease of Application	Viscosity, Shear Thinning, Yield Stress
Sagging - Brush, Spray Applications	Elasticity, Structure Recovery
Leveling - Brush Applications	Elasticity, Structure Recovery
Ribbing - Roller Applications	Viscoelastic Profile, Elasticity, Structure Recovery
Spatter	Normal Force, Elasticity
Stability / Shelf Life	Examine Linear Viscoelastic Region (G' versus Time), Resistance to Creep
Flowability	Viscosity at a given Temperature
Flowability	Viscosity at a given Temperature
Stability / Shelf Life	Examine Linear Viscoelastic Region (G' vs Time), Resistance to Creep
Phase Separation	Change of Structure with Time
Gelation	Gain of Structure, Elasticity
Product Consistency / Texture	Viscosity and Viscoelastic Behavior
Pourability & Dispensing under Pressure	Viscosity, Shear Thinning
Stability / Shelf life	Examine Linear Viscoelastic Region (G' vs Time). Resistance to Creep
Phase Separations	Change of Structure with Time
Application to Skin / Skin Feel	Viscosity, Shear Thinning, Structure Breakdown / Recovery
Gelation	Gain of Structure, Elasticity
Pourability, Pumping (in Plant)	Viscosity, Shear Thinning
Stability / Shelf life	Examine Linear Viscoelastic Region (G' versus Time), Resistance to Creep
Pouring, Pumping	Viscosity at a Given Temperature
Slip Casting, Casting performance	Viscosity, Yield Stress, Structure Changes
Oils - Pouring, Pumping	Viscosity at a Given Temperature, Compliance
Effects of Modifiers	Viscosity, Shear Thinning, Structure Changes
Outwaxing of Crude Oils	Viscosity at a Given Temperature
Greases / Lubes - Composition, Structure	Structure and Viscosity Changes
Mixing & Lubrication	Viscosity Profile, Yield Stress, Compliance, Structure Changes

Specific Examples

The following examples show the wide range of information routinely obtainable on the flow and structural properties of materials by the use of appropriately configured AR 1000 and AR 500 Rheometers. Details of these and other measurements can be obtained from your TA Instruments Representative.

Figure 1. TA Rheometers generate a flow curve by measuring shear rate under a ramped shear stress. The data provides information on yield stress, viscosity, shear thinning and thixotropy as well as correlations to real world processes (e.g. pumping, stirring and extrusion). Information on sample breakdown and recovery can lead to more cost effective manufacturing processes. Figure 1 shows a generalized flow curve for dispersions, together with relevant shear rate ranges for various processes and applications. Most dispersions follow the general shape of this curve. A high zero shear viscosity plateau is linked to a lower infinite shear viscosity plateau by a shear thinning power law region. Simple techniques (e.g. falling ball, U-tube capillary) and spindle viscometers only measure a point or small part of the curve, usually in the power law region. If measurements are not made in the relevant part of the flow curve, predictions of process and product performance can be in error. The shape of the flow curve is also critical in understanding and predicting product performance. TA Rheometers have the torque range to measure at both ultra low and high shear rates and are often able to describe the shape of the entire viscosity curve in one measurement. Rheology Advantage Software can fit experimental data to a wide variety of the latest theoretical models.

Figure 1.
Flow Curve For Dispersions

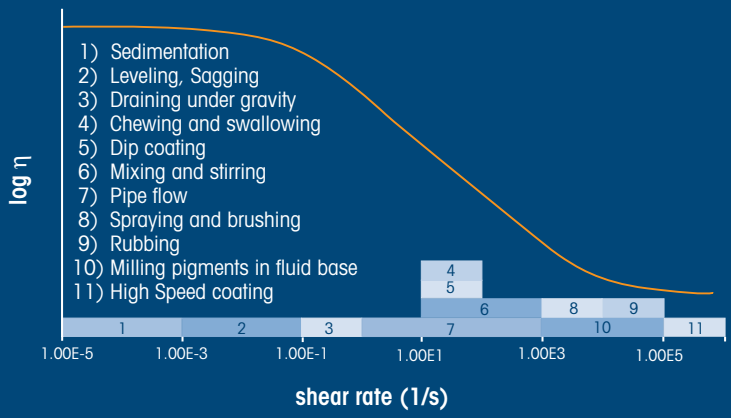


Figure 2 shows a generalized flow curve for a polymer melt together with shear rate ranges that are related to processes that a product may experience during production or where information on its molecular structure may be determined. A polymer's molecular weight greatly influences its viscosity while its molecular weight distribution and degree of branching affect its viscosity shear rate dependence. These differences are most apparent at low shear rates that cannot be attained by other devices such as melt flow index or capillary rheometry. In addition to general model fitting, Rheology Advantage software can be used to determine a molecular weight based on a measured zero shear viscosity and its internal library of K values for most common polymers. For a given polymer, comparisons can be made to study the effect of fillers, plasticizers and other additives. The range covered in a single flow test is often limited due to melt instability and fracture at modest shear rates. However, the curve can be extended well into the power law region by taking oscillatory data and applying the Cox-Merx Rule to the complex viscosity data, and if necessary can be further extended by applying the time temperature superposition principle.

Figure 2.
Flow Curve For Polymers

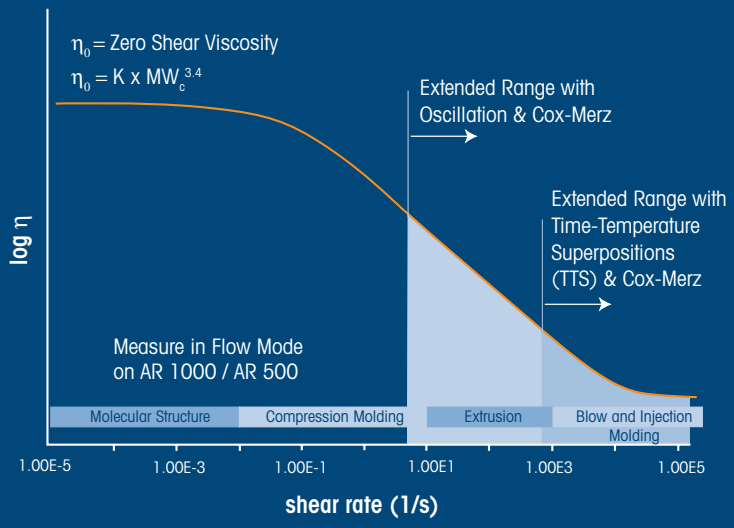


Figure 3. In a creep recovery test, a constant stress is applied to the sample and the resulting strain is measured with time. The stress is then removed and the recovery (recoil) strain is monitored also with time. If the stress is small the sample response is linear. Creep is a powerful and sensitive technique for characterizing the viscoelastic response of a material. Many industrial dispersions (suspensions and emulsions) are structured, viscoelastic fluids. Often viscosity alone cannot predict their processability and performance because elastic response can dictate the latter. Figure 3 shows the results of creep tests performed on good and bad paint samples, with the former being reported to produce a smoother finish upon drying with less evidence of brushmarks. The results show the power of creep for measuring the differences in viscoelastic structure of the two samples under the same conditions. The good sample undergoes a larger displacement than the bad sample indicating lower viscosity. In the recovery zone, it also shows a higher degree of recoil indicating that it is more elastic than the bad sample. Mathematical modeling of creep data in software is a standard feature of TA Rheometers.

Figure 4.
Creep Recovery on Polymer Melt

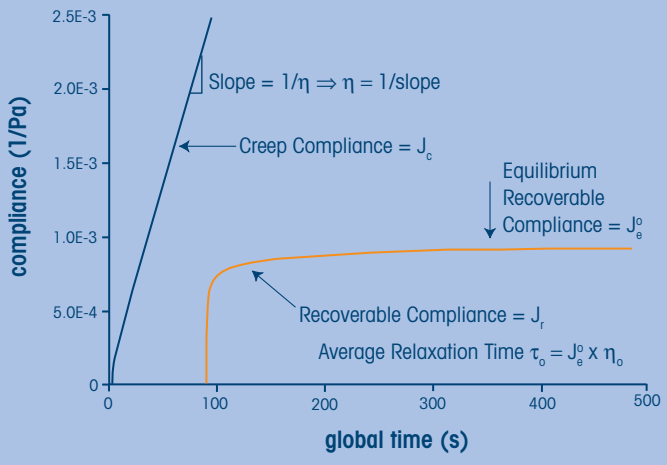


Figure 5. The oscillation mode allows the measurement of key viscoelastic parameters (G', G", η^*, tan delta, etc) as a function of stress (torque), strain, frequency, temperature or time. Figure 5 shows that structure and stability comparisons of dispersions can be easily made using oscillation stress sweep. The plots provide a direct determination of each dispersion's linear viscoelastic region (LVR), the length of which determines the stability of the dispersion. The data also shows that while one sample may have a higher modulus than another, it does not necessarily correspond to a more stable structure under the same stress conditions. Figure 5 also shows that it is possible to predict the relative differences in dispersions as they undergo structure breakdown.

Figure 3.
Creep Curves of Paint Samples

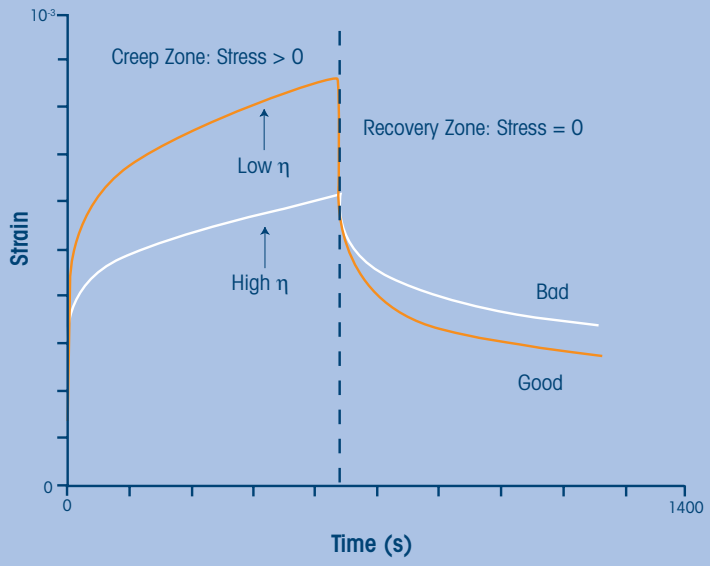


Figure 4. Creep is also a valuable method for studying the viscoelastic properties of polymer melts. In this test, it is useful to plot the creep and recoverable compliance, which are parameters calculated from the creep strain, the recoil strain and the stress applied in the creep zone (See Figure 4). Two important linear viscoelastic properties, which are crucial in flow behavior and give valuable information on molecular structure are the zero shear viscosity (η_o) and the equilibrium recoverable compliance (J_e^o). Figure 4 shows that η_o is calculated from the equilibrium slope of the creep compliance curve (J_c), while J_e^o is the value of the creep compliance curve (J_r) at full sample recoil. These parameters are greatly influenced by the molecular structure of the polymer (MW, MWD, branching). Creep recovery is also the most sensitive rheological test for detecting long chain tails, which can cause difficulty in polymer processing. In addition, the product of η_o and J_e^o is a measure of the average relaxation time of the polymer, another valuable parameter in understanding polymer performance.

Figure 5.
Oscillation Stress Sweeps on Ink Dispersions

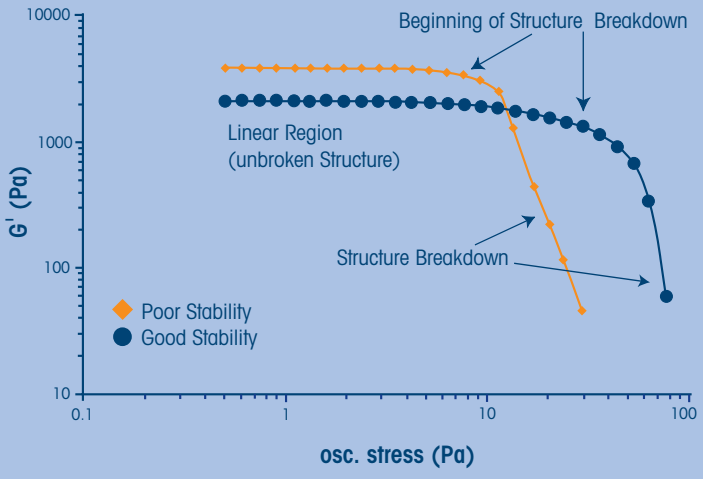


Figure 6. The linear viscoelastic properties of polymer melts are most commonly studied in oscillation mode. Figure 6 shows the storage modulus (G') and loss modulus (G") response as a function of frequency for a typical high molecular weight polymer melt. As polymer melts are viscoelastic, the mechanical response will be time dependent. Low frequencies correspond to long times. The response of the dynamic moduli shown here spans many orders of magnitude. This type of response is called a master curve and is obtained by scanning the material over a wide temperature range and applying the time-temperature-superposition (TTS) principle. The glassy region shows shortest time response and the terminal region shows the longest time response. The linear viscoelastic response of a polymer melt yields valuable information about the molecular structure such as molecular weight and molecular weight distribution. The magnitude and shape of the G' and G" curves will depend on the molecular structure of the polymer. A single frequency sweep test can be conducted to QC incoming raw material or a master curve can be generated to finger print the molecular structure of the material. In addition to the storage and loss modulus, other material parameters such as complex and dynamic viscosity, and material damping are simultaneously measured.

Figure 6.
Frequency Sweep: Material Response

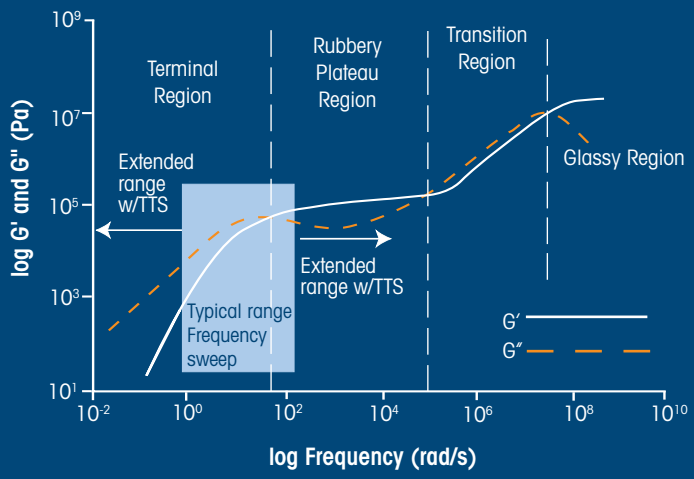
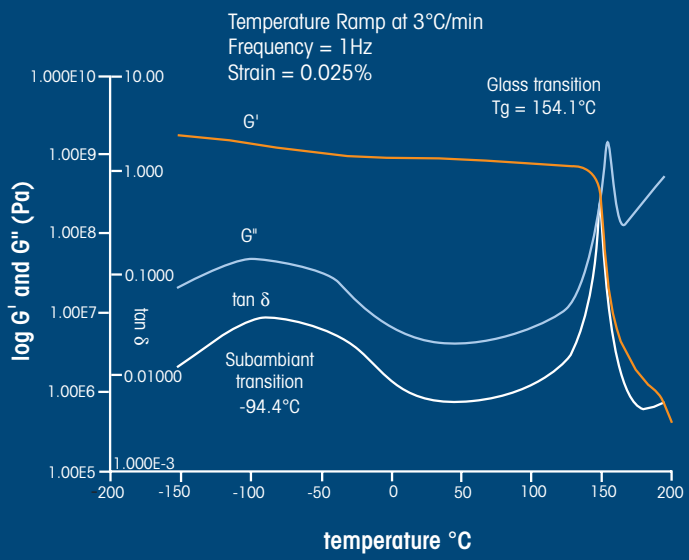


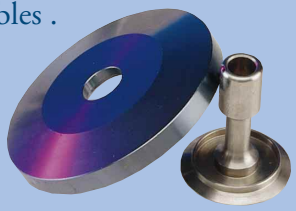
Figure 7. The ability to characterize the solid properties of polymers in torsion is a feature of the TA Instruments rheometers. Polymers are widely used because of their desirable mechanical properties and economical cost. In most applications, the mechanical properties are considered the most important of all properties. Therefore, those working with polymers need to have at least some basic knowledge of the mechanical behavior (strength, modulus), in addition to an understanding of how the mechanical behavior can be modified by varying structural factors such as molecular weight, crystallinity, and cross-linking. The linear viscoelastic response of a solid polymer yields valuable information about the molecular structure. The magnitude and shape of the storage modulus (G'), loss modulus (G"), and damping (tan δ) curves will depend on the chemical composition and molecular structure of the polymer. Figure 7 shows a typical temperature ramp scan from –150°C to 200°C, at a frequency of 1 Hz (6.28 rad/s) and a ramp rate of 3°C/min. for a polycarbonate. Transitions or relaxations of molecular segments in the polymer are observed as step changes in the storage modulus and as peaks in the loss modulus and damping.

Figure 7.
PC - Polycarbonate



Measurement Geometries

TA Instruments offers a wide range of measurement geometries (cones, parallel plates, concentric cylinders). Most are available with solvent traps and covers. Materials include stainless steel, hard anodized aluminum, acrylic and titanium. For elevated temperature Peltier Plate operation, a series of stainless steel geometries with composite heat breaks are available. Disposable/reusable cones and plates are available for applications involving thermosets. TA Instruments can supply custom geometries using a variety of materials to meet special requirements. Details of our standard geometries, including dimensions, and minimum / maximum shear rates are shown in the tables .



Software

Easy to use, intelligent, and automated. TA Instruments has been writing rheology software for over 15 years. Rheology Advantage 32-bit software offers flexible experimental design, and rapid, accurate data analysis. See our Rheology Advantage software brochure for more details.

Parallel Plate Geometries

Diameter	Shear Rate min, 1/s	Shear Rate max, 1/s
8mm	4.000E-07	4.00E2
20mm	1.000E-06	1.00E3
25mm	1.250E-06	1.25E3
40mm	2.000E-06	2.00E3
60mm	3.000E-06	3.00E3

Note: Shear rates calculated @ gap of 1mm. Shear rates will vary with gap.

Cone and Plate Geometries

Cone Angle	Shear Rate min, 1/s	Shear Rate max, 1/s
20 mm 0.5°	1.146E-05	1.146E4
20 mm 1°	5.730E-06	5.730E3
20 mm 2°	2.865E-06	2.865E3
20 mm 4°	1.432E-06	1.432E3
40 mm 0.5°	1.146E-05	1.146E4
40 mm 1°	5.730E-06	5.730E3
40 mm 2°	2.865E-06	2.865E3
40 mm 4°	1.432E-06	1.432E3
60 mm 0.5°	1.146E-05	1.146E4
60 mm 1°	5.730E-06	5.730E3
60 mm 2°	2.865E-06	2.865E3
60 mm 4°	1.432E-06	1.432E3

Concentric Cylinders

Geometery	Shear Rate min, 1/s	Shear Rate max,1/s
Din (Conical)	1.452E-06	1.452E3
Recessed Rotor	1.452E-06	1.452E3
Double CC	5.314E-06	5.314E3

TA Instruments
Thermal Analysis & Rheology

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