TA Instruments

AR Rheometer Temperature Systems and Accessories

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AR Series

All AR Series temperature systems and accessories are designed with superior performance and ease-of-use in mind. The innovative design and features of these accessories help make them the most popular line of rheometers in the world. Only TA Instruments AR Series Rheometers offer the convenience and versatility of Smart Swap™ temperature control options and accessories. Smart Swap options are attached to the instrument on its unique magnetic base. Once attached, the instrument automatically detects and configures the system for operation.

Peltier Plate Temperature Systems

The most common temperature control option for the AR Series rheometers is the Peltier Plate. Over 20 years ago, TA Instruments was the first rheometer supplier to introduce Peltier Plate temperature control. TA Instruments understands the value of Peltier heating and cooling for rheological measurements and has adapted this core technology to meet the widest range of material applications. It is now the highest performing, most versatile, and best accessorized Peltier Plate Temperature System available.

Available Peltier Plates

Three Smart Swap™ Peltier Plate options are available for the AR Series. The standard Peltier Plate, a stepped Peltier Plate, and a stepped disposable Peltier Plate. These options bring the convenience of Peltier temperature control to a much broader range of rheological applications. The standard Peltier Plate has a continuous 80 mm diameter hardened chrome surface. The stepped Peltier Plate can accommodate up to 25 mm diameter lower plates, available in stainless steel with flat and crosshatched surfaces. For special applications, plates can be made out of other materials such as titanium. This option also allows for samples to be prepared and/or conditioned outside of the rheometer and installed when ready for testing. The stepped disposable Peltier Plate is ideal for thermoset curing, or other single use applications and is compatible with standard disposable plates. The lower plates are held firmly with two locking screws as shown in the figure below.

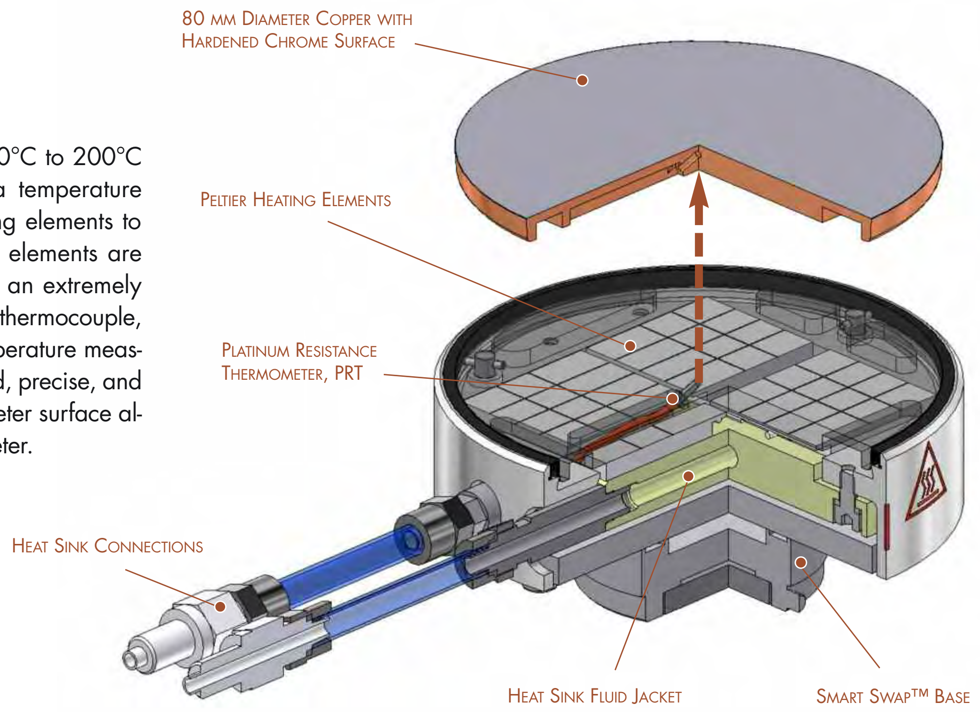
Standard Peltier Plate

Stepped Peltier Plate

Stepped Disposable Peltier Plate

Technology

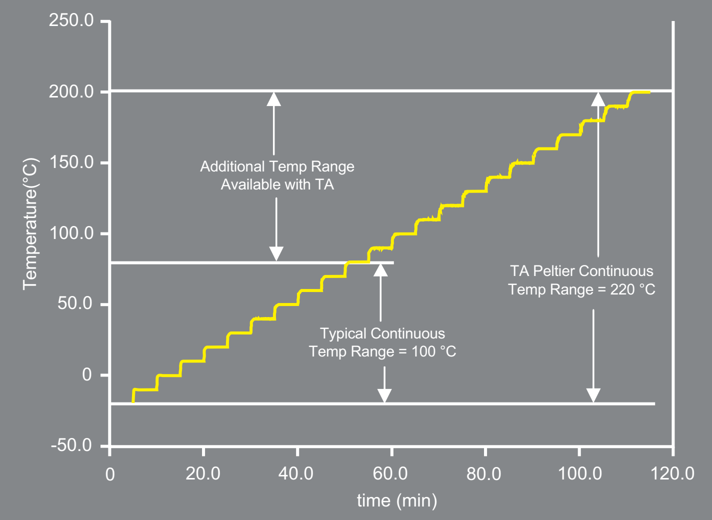
AR Series Peltier Plates offer a temperature range of –40°C to 200°C with typical heating rates of up to 50°C/min and a temperature accuracy of 0.1°C. They incorporate four Peltier heating elements to cover an 80 mm diameter plate surface. These Peltier elements are placed directly in contact with a thin copper disc with an extremely rugged, hardened chrome surface. A platinum resistance thermocouple, PRT, is placed at the exact center ensuring accurate temperature measurement and control. The unique design provides for rapid, precise, and uniform temperature control over the entire 80 mm diameter surface allowing standard geometries to be up to 60 mm in diameter.



Performance

Peltier heating devices require a circulating fluid heat exchange medium (heat sink), typically water. Only the unique design of TA Instruments' Peltier Plate can provide 220°C of continuous temperature range with a single heat sink temperature. The data in the figure below shows temperature steps from –20°C to 200°C at 10°C increments. Typical continuous temperature range of Peltier systems at a single heat sink temperature is 100°C. TA Instruments' design more than doubles the actual useable temperature range during any test.

AR Peltier Plate Temperature Steps Over 220°C Range



Features and Benefits

- Unique Smart Swap Technology
- Wide Temperature Range: –40°C to 200°C
- Widest continuous temperature range
- Accurate Temperature Control: +/- 0.1°C
- Hard chrome surface
- Standard, stepped, and disposable models
- Plates and Cones up to 60 mm in diameter
- Large variety of geometry materials and types
- Fully accessorized
  - Extremely efficient solvent trap
  - Smooth, crosshatched, and sandblasted covers
  - Purge Gas cover
  - Insulating Thermal cover
  - Camera viewer option
  - Immersion cell

Peltier Plate Geometries

The Peltier Plate system can be used with Cone and Plate and Parallel Plate geometry types. They are available in a variety of sizes, cone angles and material types. Custom geometries of non-standard sizes, materials, and surface finishes, such as sandblasted or Teflon®-coated, are available upon request.

General Connection

AR 2000ex and AR 1500ex rheometers use the standard geometry coupling system. The AR-G2 rheometer uses TA Instruments' unique Smart Swap™ geometries with automatic recognition.

Standard Geometry Dimensions

Peltier Plate Geometries are available in 8 mm, 20 mm, 25 mm, 40 mm, and 60 mm diameters. Upper Cone geometries are readily available in 0.5°, 1°, 2°, and 4° cone angles. Non-standard diameters and cone angles are available upon special request. Changing diameter and cone angle impact the measurable range of stress and strain or strain rate.

Materials of Construction

Peltier Plate geometries come standard in the following materials:

- **Stainless Steel:** Rugged, very good chemical resistance for highly basic or acidic materials.
- **Stainless Steel with Composite Heat Break:** Same properties as stainless steel with added benefit of composite heat break, which insulates upper geometry when controlling temperatures away from ambient.
- **Hard Anodized Aluminum:** Excellent thermal conductivity, low mass, fair chemical resistance.
- **Acrylic:** Very low mass, Max Temp 40°C, optically clear for sample viewing. Note: Not available for AR-G2.
- **Titanium:** Low mass, excellent chemical resistance.

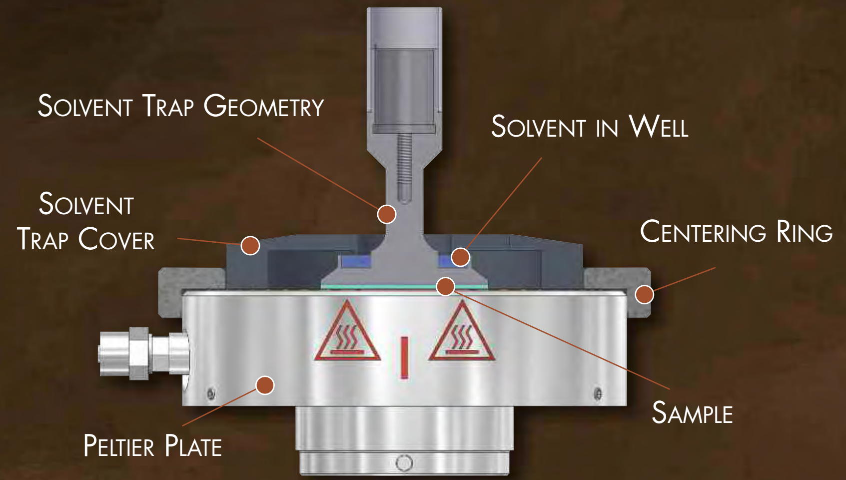
Peltier Plate Standard Geometry Types

Peltier Cone and Peltier Plate Geometries are available in three basic types. They include geometries without solvent trap, geometries with insulating composite heat break, and geometries with solvent trap. Heat break geometries are available in stainless steel only. Solvent trap geometries are designed for use with the solvent trap system discussed separately. The figure below shows a comparison of stainless steel 40 mm geometry types.

No Solvent Trap — With Solvent Trap — With Solvent Trap & Composite Heat Break

Peltier Solvent Trap

Many samples are prone to drying due to solvent evaporation during testing. The Solvent Trap cover and Solvent Trap geometry work in concert to create a thermally stable vapor barrier, virtually eliminating any solvent loss during the experiment. The geometry includes a well that contains very low viscosity oil, or even the volatile solvent present in the sample. The Solvent Trap cover includes a blade that is placed into the solvent contained in the well without touching any other part of the upper geometry. The Solvent Trap sits directly on top of the Peltier Plate surface and an insulating centering ring insures perfect placement for quick and easy sample loading.



Purge Gas Cover

The purge gas environmental cover is a hard-anodized aluminum two-piece split cover with 6 mm diameter compression fittings. An insulating location ring insures precise and easy location of the cover. This cover is ideal for purging the sample area with nitrogen to prevent condensation during experiments performed below room temperature or with a humidified purge to keep a sample from drying.

Insulating Thermal Cover

The thermal insulation cover is constructed of an Anodized Aluminum core surrounded by an insulating cover. The aluminum core conducts heat to the upper geometry providing uniform temperature throughout the sample. Recommended for use over a temperature range of -10°C to 90°C, with samples not susceptible to drying such as oils, caulk, epoxy, and asphalt binder. Heat break geometries are recommended for use with the cover.

Peltier Immersion Ring

The Peltier Plate immersion ring allows samples to be measured while fully immersed in a fluid. The immersion ring is compatible with all Peltier Plate models and is easily attached to the top of the Peltier Plate. A rubber ring provides the fluid seal. This option is ideal for studying the properties of hydrogels.

Peltier Plate Covers

A variety of Peltier Plate covers are available for applications that can harm the chromium surface of the plate or for samples that exhibit slip during testing. They are available in stainless steel, hard-anodized aluminum and titanium. Crosshatched and sandblasted Peltier covers are used to eliminate sample slippage effects. Covers are compatible with solvent trap.

Peltier Plate Camera Viewer

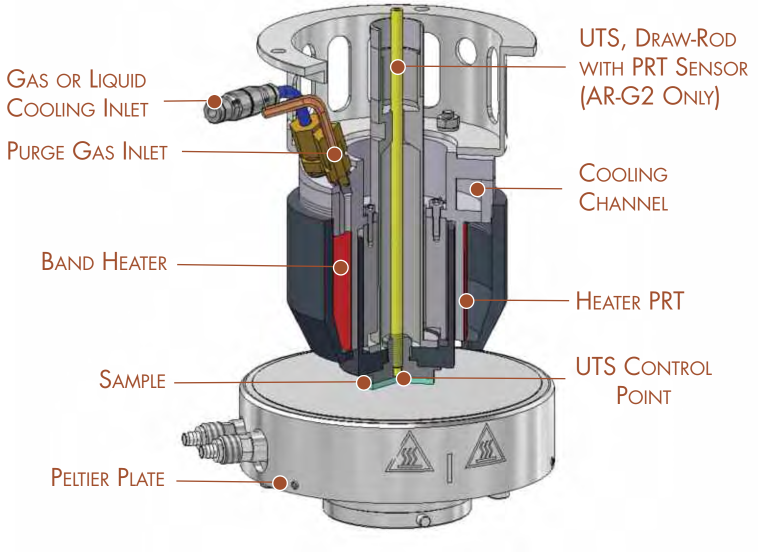
The camera viewer is used in conjunction with streaming video and image capture software. Real time images can be displayed in the software and an image can be stored with each data point for subsequent viewing during data analysis. The camera viewer is perfect for long experiments with unattended operation as visual inspections of data integrity.

Upper Heated Plate for Peltier Plate (UHP)

Lower Peltier plates have a maximum temperature of 200°C. However, vertical temperature gradients when heating from only the bottom can become significant at temperatures above 50°C, leading to errors in the absolute rheological data. The UHP is compatible with all Peltier Plate models and provides upper plate temperature control. The UHP features 8, 25, and 40 mm diameter cones and plates and a maximum temperature of 150°C. The lower temperature can be extended using a variety of flexible liquid and gas cooling options. (Note: To extend the upper heater temperature range to 200°C, see electrically heated plates option).

Technology

The UHP sets a new standard in non-contact heating with a design that delivers optimum heat transfer with minimum sample thermal equilibration time. An electrical band heater is in direct contact with an aluminum thermal transfer block. At the top of the block is a cooling channel for both liquid and gas cooling medium. A patented[(1)] geometry design incorporates a cup-shaped vertical heat spreader, surrounded by the thermal transfer block. This FEA optimized design provides a large surface area for efficient heat transfer to the plate surface. Unlike traditional designs, the UHP system and geometry move in concert ensuring gap independent heat transfer to the sample. Also unique to the UHP are calibration and modeling routines that match upper and lower temperatures and heating rates. This ensures the sample is heated uniformly from both sides and allows for true temperature ramps on materials. The AR-G2 takes the UHP to a new level with patented Active Temperature Control, ATC, for active measurement and control of the upper plate temperature, eliminating the need for temperature offset calibrations. This technology and its benefits are discussed in the next section.

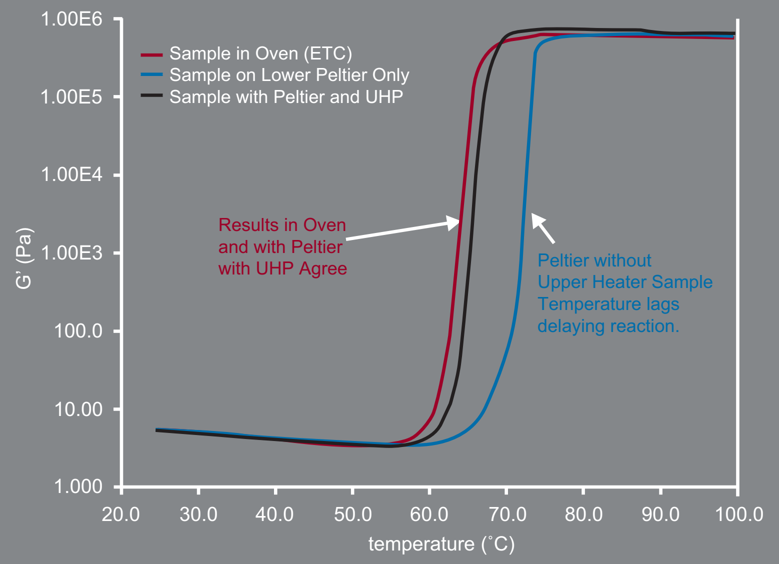


(1) Patent # US#7168299

Features and Benefits

- Unique Smart Swap™ option
- Wide temperature range: -30°C to 150°C
- AR-G2 patented Non-Contact upper temperature sensor
- Compatible with standard, stepped, and disposable Peltier Plate systems
- Modeled for optimum heat transfer with minimum sample thermal equilibration time
- Sample gap independent heat transfer
- True controllable heating rates up to 15°C per minute
- Sample trimming and plate removal tools
- Gas purge port and environment cover
- Disposable Plate system
- Flexible cooling options

Advantage of UHP with Lower Peltier



Plastisol Sample Evaluation with Upper Heated Plate

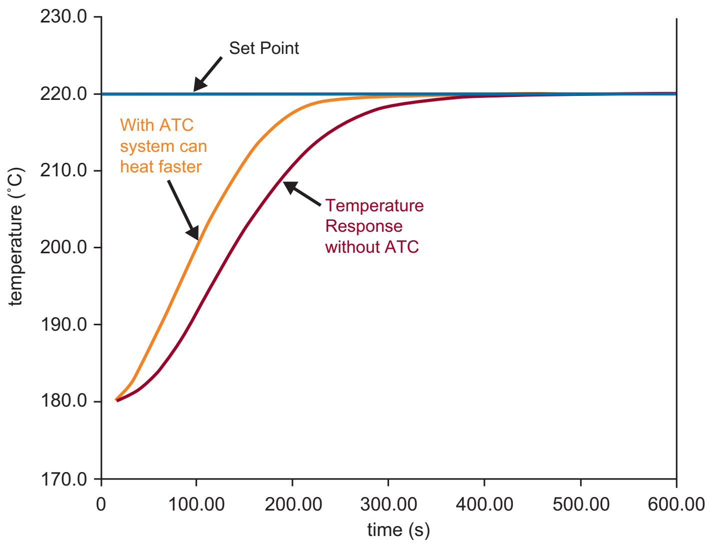
A plastisol is a mixture of resin and plasticizer that can be molded, cast, or formed into a continuous film by applying heat. Plastisols are used in applications such as screen-printing on fabrics and ink printing where the material is hardened at moderately low temperatures. The graph shows the value of the Upper Heated Plate (UHP) for eliminating sample temperature gradients. An oscillatory time sweep was conducted on a plastisol using three temperature system configurations. A combined convection-radiation oven, ETC, a lower Peltier Plate only, and a lower Peltier Plate with UHP. The figure shows when the sample is surrounded by heat from the top and bottom, as in the ETC and UHP, the hardening temperature, observed as a sharp increase in G′, occurs at approximately 60°C. However, when heating the sample using only the lower Peltier Plate, the sample temperature lags behind the heating profile due to the vertical gradient, making the hardening point appear to be approximately 70°C.

Patented Active Temperature Control (ATC)

ATC Performance Advantage

Active Temperature Control, ATC, is patented technology that enables active measurement and control of non-contact upper heating systems. The benefit of this technology is that it provides optimized temperature control when compared to traditional passive non-contact heating systems that require offset calibrations. The improved responsiveness of ATC is demonstrated on a polymer melt and shown in the figure to the right. A temperature step was performed on the Electrically Heated Plates, EHP, from 180 to 220°C with and without ATC. The data with ATC exhibit a faster heating rate as observed by the steeper slope, as well as a more rapid arrival to the final temperature of 220°C.

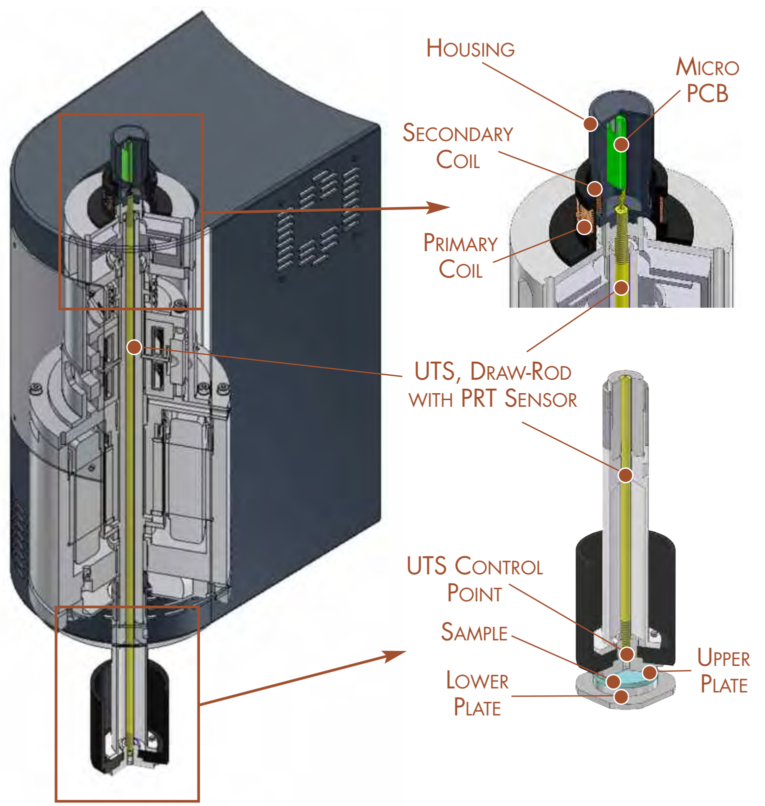
Temperature Response Improvement with ATC



ATC Technology

The AR-G2 Upper Heated Plate (UHP) and Electrically Heated Plate (EHP) temperature systems incorporate the new patented[1] non-contact temperature sensor for active measurement and control of the upper plate temperature using a special draw rod. A draw rod is a thin steel cylinder that passes through the center of the rheometer head to couple the geometry and motor. This special draw rod is called the upper temperature sensor or UTS. The UTS houses a micro PCB and a secondary coil in the knob, and a Platinum resistance thermocouple with the sensing point at the tip. As shown in the figure below, standard draw rod connections are made at the top of the geometry shaft, while the UTS passes completely through the geometry connecting directly into the upper rotating plate. The PRT senses temperature at the tip of the cone or plate geometry and the signal is transmitted from the secondary coil to a primary coil in the rheometer head assembly. This technology provides, without any mechanical contact, a temperature read-out for controlling the actual upper plate temperature. Together with the PRT in the lower plate, real-time control of both plates is possible allowing temperature to be changed in both at the same rate. This eliminates vertical sample gradients and the need for complex calibration procedures and offset tables resulting in a more responsive and accurate system.

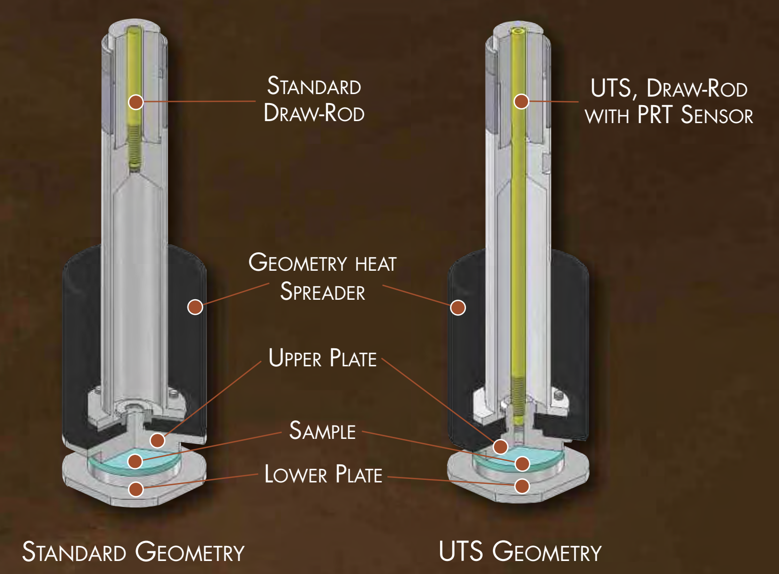
(1) Patent # 6,931,915



Features and Benefits

- Available for Upper Heated Plate (UHP) and Electrically Heated Plate (EHP) Temperature Systems
- Only active temperature measurement and control of non-contact heating system available
- Significant temperature control advantages
- Temperature known rather than inferred
- No need for offset calibrations and tables
- True temperature ramp capability
- Faster temperature response compared to traditional non-contact systems

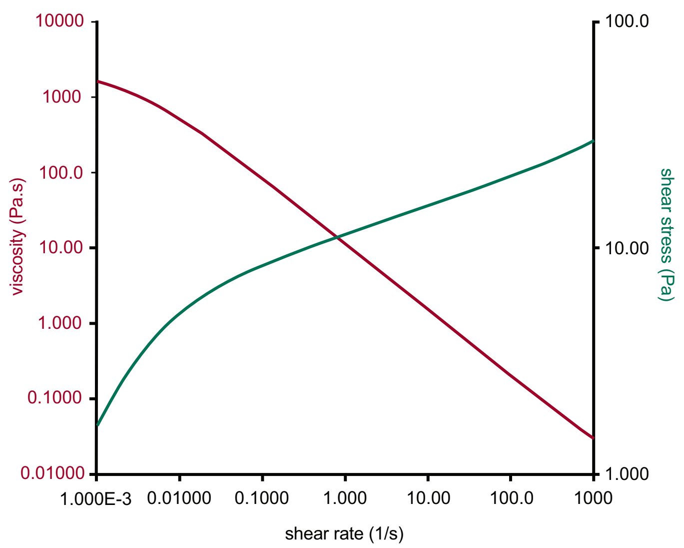
Comparison of Standard and UTS Connection



Concentric Cylinder Temperature Systems

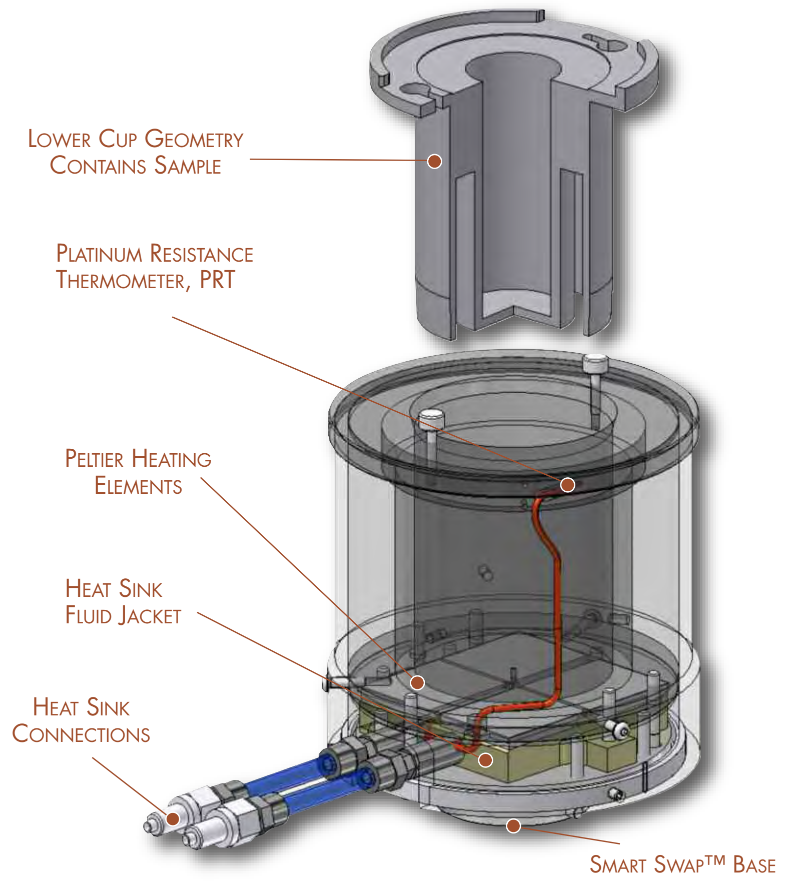
The AR Series Peltier Concentric Cylinder Temperature System combines the convenience of Smart Swap™ and Peltier heating technology with a wide variety of cup and rotor geometries. Concentric cylinder geometries are commonly used for testing low viscosity fluids, dispersions or any liquids that are pourable into a cup. Examples of materials suitable for Concentric Cylinders include low concentration polymer solutions, solvents, oils, drilling mud, paint, varnish, inkjet ink, ceramic slurries, pharmaceutical suspensions such as cough medicine and baby formula, foams, food products such as juices, thickeners, dairy products such as milk and sour cream, salad dressings, and pasta sauce.

Xanthan Gum Solution in Concentric Cylinder



Flow Curve on Xanthan Gum

Concentric Cylinder geometries are useful for gathering viscosity flow curve information over a wide range of shear rates. An example is shown in the figure for a Xanthan Gum solution. Five decades of viscosity are easily obtained over six decades of shear rate. This system is also a powerful alternative to parallel plate or cone and plate geometries for materials with limited stability or prone to edge failure or rapid solvent evaporation.



Technology

The AR Series Peltier Concentric Cylinders offer a temperature range of –20°C to 150°C, with a maximum heating rate up to 13°C/min. Four Peltier heating elements are placed in intimate contact with a lower cup geometry held in place by an insulated jacket. The unique design of the lower geometry provides fast and efficient heat transfer up the walls of the cup. A platinum resistance thermocouple, PRT, is placed close to the top of the cup ensuring accurate temperature measurement and control. Maximum controllable heating rate will depend on heat sink fluid temperature, circulator flow rate and cooling/heating capacity, and viscosity of heat sink fluid.

Features and Benefits

- Unique Smart Swap™ technology(1)
- Wide temperature range: -20°C to 150°C
- Peltier temperature control for fast heating and cooling
- Popular DIN standard, Recessed End and Double Gap Options
- Geometries available in Stainless Steel and Hard Anodized Aluminum
- Wide variety of cup diameters
- Impeller and Vane geometeries for preventing settling and slipping, and handling of large particles
- Pressure Cell
- Torsion Immersion
- Special geometries available upon request

(1) Patent # 6,588,254

Concentric Cylinder Geometries

Traditional and specialty Concentric Cylinder geometries are available in a variety of sizes and material types. Geometries are readily available in hard-anodized aluminum and stainless steel, and can be made from other materials, such as titanium, upon special request.

Cup and Rotor Systems

The standard Peltier Concentric Cylinder geometries include a standard cup radius of 15 mm, configured with either a Recessed End or DIN Rotor. Both rotors have a radius of 14 mm and height of 42 mm. The double gap concentric cylinder has an additional shearing surface over single gap providing lower stress and higher sensitivity for extremely low viscosity solutions.

Double Gap Rotor & Cup

DIN Rotor & Standard Cup

Special Cups and Rotors

Specialty geometries include various vanes and starch pasting impeller rotors, as well as large diameter and grooved cups. These special concentric cylinder geometries are very valuable for characterizing dispersions with limited stability, preventing error from slip at the material/geometry interface, and for bulk materials with larger particulates. Vane geometries are available in both a 14 mm and 7.5 mm radius. The large diameter cup has a radius of 22 mm. Impeller rotor and cup, with 18.5 mm radius, keep a sample mixed or particles suspended during shearing. The Conical Rotor provides more traditional rheometry measurements with the Impeller cup.

Starch Pasting Impeller & Cup

Large Gap Vane Rotor & Large Diameter Cup

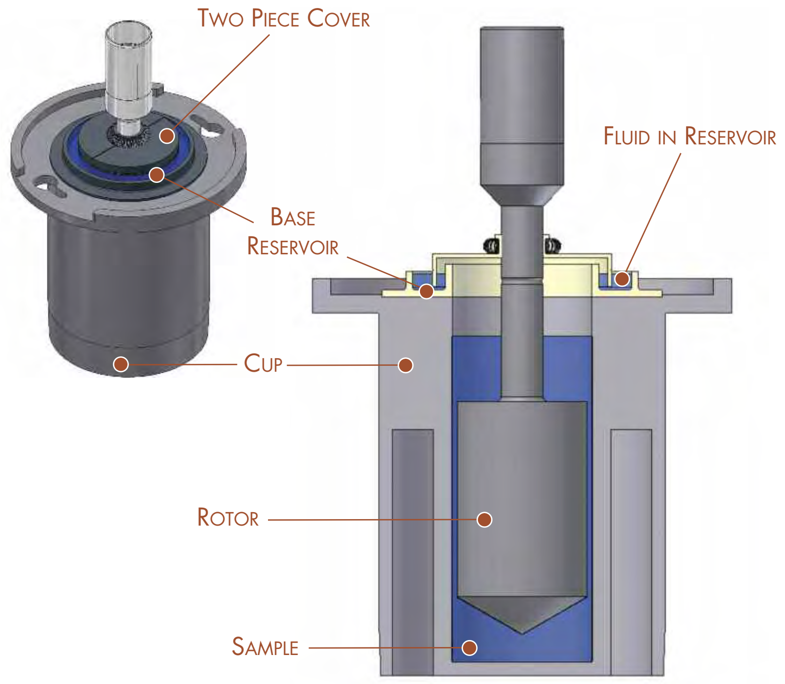
Vane Rotor & Grooved Cup

Concentric Cylinder Cup and Rotor Compatibility Chart

Cup \ Rotor	DIN	Recessed End	Starch Impeller	Conical	Vane	Wide Gap Vane	Double Gap
Standard Cup (rad=15 mm)	•	•			•	•	
Large Diameter (rad=22 mm)	•	•	•	•	•	•	
Starch Pasting (rad=18.5 mm)	•	•	•	•	•	•	
Grooved Cup					•	•	
Double Gap							•

Concentric Cylinder Solvent Trap Cover

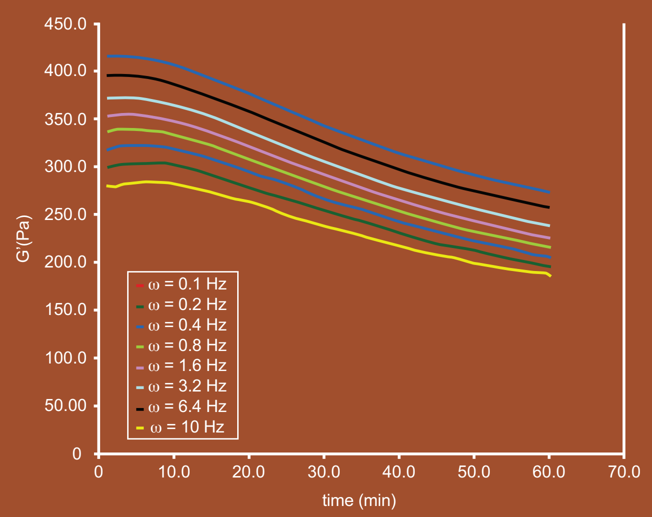
An optional solvent trap is available for the Peltier Concentric Cylinder. It includes a base reservoir and a two-piece cover that is mounted to the shaft of the rotor. The solvent trap provides a vapor barrier to seal the environment inside the cup and prevents solvent evaporation.



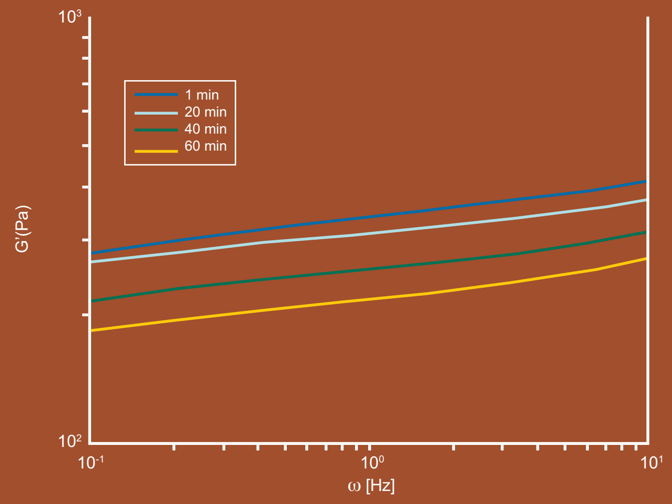
Characterization of Foam with Vane Rotor

The Figures below show an example of the time and frequency dependent response of a foam shaving cream characterized using a standard cup and vane geometry. The structure of shaving foam has a limited lifetime, or limited stability. The vane geometry minimizes shearing stress that occurs during loading in the gap with standard rotor, keeping the delicate foam structure intact for testing. A wide range of structural information can be captured very quickly using Multiwave characterization on the AR Rheometer. The figure to the left shows a decay in storage modulus G′ as the structure of the foam breaks down with increasing time. Using multiwave the data are simultaneously collected over a wide range of frequencies. The data can be plotted as frequency sweeps at increasing time. These data show the time-dependent viscoelastic response of the shaving foam.

Multiwave Time Sweep

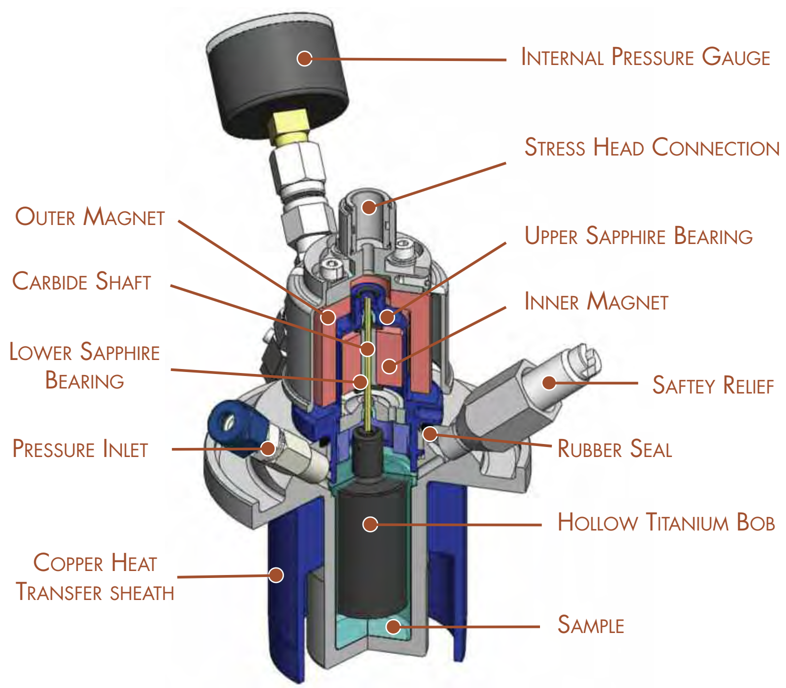


Frequency Sweeps on Foam at Different Time Intervals



Pressure Cell

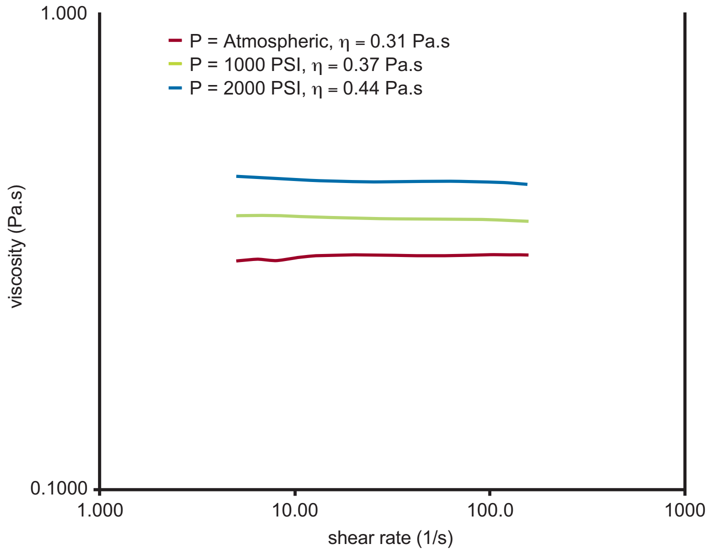
The Pressure Cell is a sealed vessel that can be pressurized up to 138 bar (2,000 PSI), over a temperature range of –10 to 150°C. It can be used either in self-pressurizing mode, in which the pressure is produced by the volatility of the sample, or by externally applying the pressurization, typically with a high pressure tank of air or nitrogen gas. All necessary plumbing and gauges are included as a manifold assembly. The Pressure Cell is ideal for studying the effect of pressure on rheological properties, as well as studying the materials that volatilize under atmospheric pressure. This option is available for the AR-G2 and AR 2000ex rheometers.



Technology

The pressure cell option is used with the Peltier Concentric Cylinder jacket. At the top is an outer magnet assembly that houses strong rare earth magnets and attaches to the stress head. At the bottom is a stainless steel cup, to withstand high pressure, surrounded by a copper sheath for excellent heat transfer. Also connected to the cup are the pressure inlet, internal pressure gauge, and pressure relief valve. The rotor assembly houses the inner magnet attached to a low friction carbide shaft, supported above and below by sapphire bearings. A hollow titanium cylindrical rotor is attached to the bottom of the carbide shaft. When the upper magnet assembly is lowered over the rotor assembly, the inner and outer magnets couple, levitating the rotor assembly. The Rheometer's motor movement is then transferred to the rotor through a strong magnetic coupling to drive the cylinder inside the vessel and deform the sample. This innovative high-powered magnetic coupling and low friction bearing design allows for both steady shear and dynamic measurements.

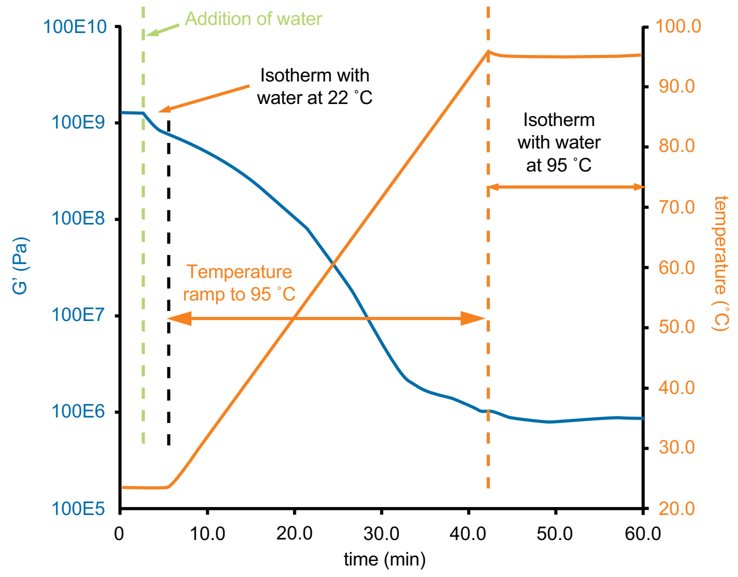
Effect of Pressure on Viscosity of Motor Oil



Effect of Pressure on Motor Oil

To understand the ability of motor oil to provide necessary lubrication under different environmental conditions, it is critical to know its viscosity over a range of temperatures and pressures. The figure above shows results of steady state flow tests conducted on automotive motor oil. The temperature was held constant at 20°C and tests were run at atmospheric pressure, 1,000 PSI (69 bar), and 2,000 PSI (138 bar) of pressure. The results show that the pressure acts to increase internal friction, as observed by the increase in viscosity.

Rheology of Pasta During Cooking



Rheology of Pasta During Cooking

The Torsion Immersion Cell can be used for various food applications such as cooking of pasta. In this example, a piece of fettuccini pasta was tested using an oscillatory time sweep test at a frequency of 6.28 rad/s and temperature of 22°C. Data were collected on the dry sample for 2.5 min. to establish a baseline storage modulus G′. Water was added after 2.5 min. and the effect of the moisture is seen immediately as a decrease in the G′. At 5 min. G′ was monitored as temperature was ramped to 95°C and held isothermally. As the pasta cooks the modulus drops about three decades and then levels out when cooking is complete.

Torsion Immersion Cell

The Torsion Immersion Cell allows rectangular bar-shaped samples to be clamped and characterized while immersed in a temperature-controlled fluid. The resulting change in mechanical properties, caused by swelling or plasticizing, can be analyzed in oscillatory experiments. This option provides a way to better understand materials under real world conditions, such as body implants in saline or rubber seals in contact with oils and solvents.

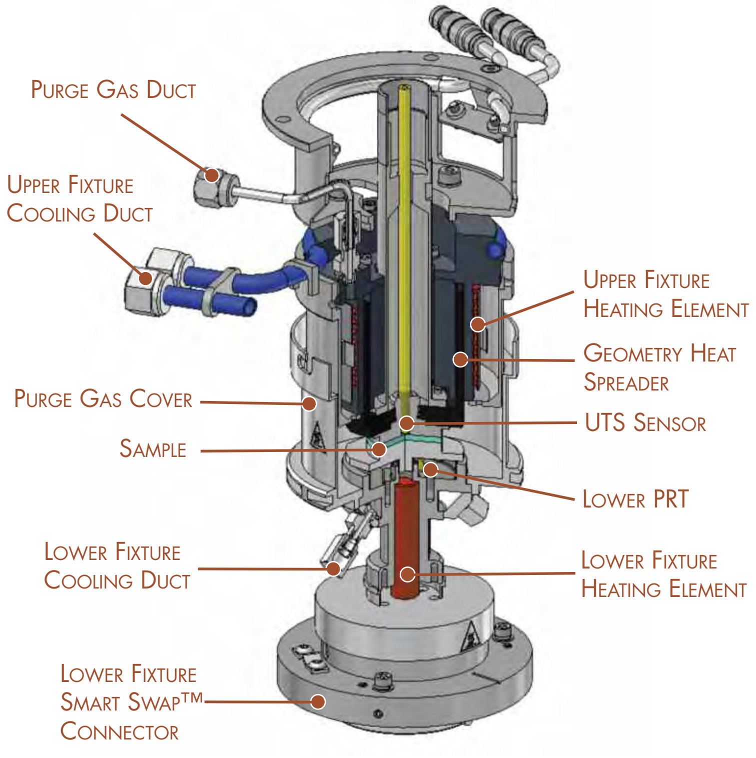
Electrically Heated Plates (EHP)

The EHP provides active heating and cooling of parallel plate and cone and plate geometries. With standard and disposable systems it's ideal for rheological characterization of polymer melts and thermosetting materials up to a maximum temperature of 400°C. The optional Gas Cooling Accessory extends the minimum temperature to –70°C. Standard features include 25 mm diameter parallel plate geometry, environmental cover, and heated purge gas. An optional clear cover is available for sample viewing and for use with the Camera Viewer option. The AR-G2 EHP offers Active Temperature Control, ATC, making it the only EHP system capable of direct temperature control of the upper and lower plates (See ATC Section for more details on this exciting technology). The Upper EHP can be used with lower Peltier Plates for temperature control to 200°C and as temperature control to 150°C for UV curing options.

Features and Benefits

- Unique Smart Swap™ option
- AR-G2 ATC with patented Non-Contact Upper Temperature Sensor
- AR-G2 patented Smart Swap geometries
- Maximum temperature of 400°C
- Optional low temperature cooling to –70°C
- Maximum heating rate of up to 30°C/min
- Controllable heating rates of 10°C/min
- Environmental cover and heated purge gas
- Modeled for optimum heat transfer with minimum sample thermal equilibration time
- Heat transfer to sample independent of gap setting
- Sample trimming and plate removal tools
- Disposable Plate System
- Optional glass cover for sample viewing and for use with camera
- Ideal for QC testing or R&D
- Upper heater compatible with all Peltier Plate systems and UV Curing options

EHP Disposable Plates



Technology

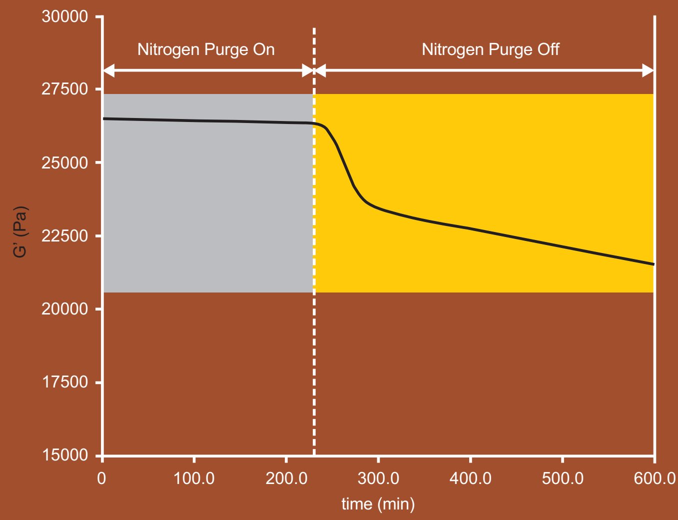
The EHP consists of independent upper and lower temperature control systems. The stationary lower assembly incorporates a cartridge-heater for direct contact heating, while it is cooled via a cooling channel surrounding the area below the lower plate. A Platinum Resistance Thermocouple, PRT, is positioned close to the plate surface for intimate control near the sample. The Upper temperature assembly sets a new standard in non-contact heating with a design that delivers optimum heat transfer. It incorporates an electrical band heater attached to an aluminum thermal transfer block and a duct for gas-cooling medium. A patented[1] geometry design incorporates a cup-shaped vertical heat spreader surrounded by the thermal transfer block. This FEA optimized design provides a large surface area for efficient heat transfer to the plate surface. Unlike traditional designs, the EHP system and geometry move together ensuring gap independent heat transfer to the sample. The AR-G2 includes Active Temperature Control, ATC, for active measurement and control of the upper plate temperature using a special Upper Temperature Sensor, eliminating the need for temperature offset calibrations (See Active Temperature Control section of the brochure for more details on this exciting technology). For samples prone to thermo-oxidative degradation, the EHP offers a heated purge and cover to create an oxyge-free environment around the sample.

(1) Patent # US#7168299

Controlling Polymer Degradation During Testing

Viscoelastic properties of polymer melts can be affected by thermal and oxidative degradation at elevated temperatures. It is important to measure rheological properties in the absence of degradation, as well as evaluating the effectiveness of stabilizing additives such as anti-oxidants. The figure shows how effective the EHP controls the environment for commercial polystyrene melt during a 10-hour time sweep experiment at 200°C. The storage modulus, G', can be seen to be very stable during the early stages of the test when the sample is purged with nitrogen. The data demonstrate the environment in the EHP is virtually oxygen-free. After about 4 hours the inert gas is shut off and the effect of presence of oxygen on the viscoelastic response is seen immediately. The polystyrene degrades as evidenced by the sharp decrease in G'.

Controlling Polymer Degradation During Testing



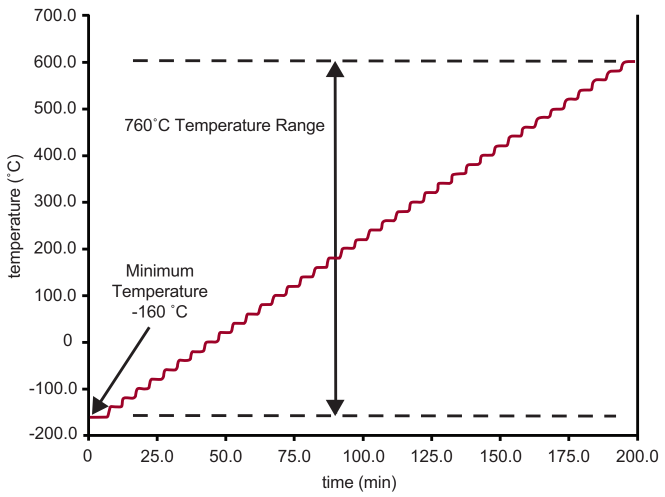
Environmental Test Chamber (ETC)

The ETC is a high temperature Smart Swap™ option available for the AR-G2 and AR 2000ex rheometers. It uses a controlled convection / radiant-heating concept and has a temperature range of –160 to 600°C with heating rates up to 60°C/min. The unique design of the ETC provides fast response and temperature stability over a continuous 760°C range as highlighted in the figure below with temperature steps of 20°C. The ETC is a very popular option for polymer applications and can be used with parallel plate, cone and plate, disposable plate, and rectangular torsion clamps for solids. Typical materials that can be tested include thermoplastics, thermosets, elastomers, caulks and adhesives, solid polymers, asphalt binder, and oils and greases.

Features and Benefits

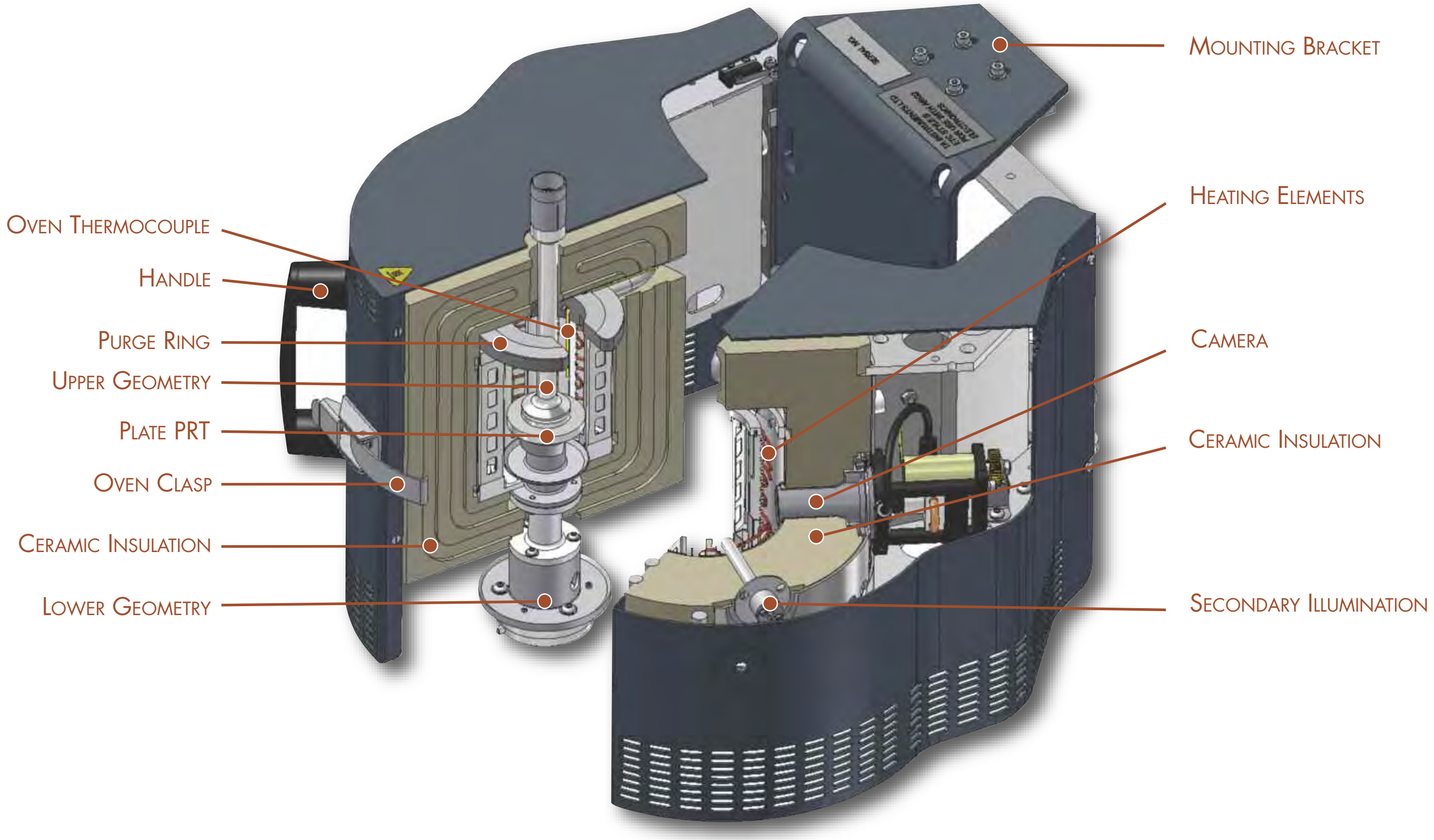
- Unique Smart Swap™ option
- No extra electronic boxes
- Combined convection and radiant heating design
- Wide temperature range: -160°C to 600°C
- Maximum heating rate of 60°C/min
- Liquid nitrogen option connects directly to bulk source
- Wide variety of stainless steel plates, cones, crosshatched, and disposable geometries
- Optional built-in Camera Viewer
- Melt rings for thermoplastic pellet samples
- Die punch for molded plaques
- Sample cleaning and trimming tools
- Extensional rheology measurements with SER2 Universal Testing Platform

ETC Temperatiure Range Performance



ETC Technology

The Smart Swap™ ETC is a "clam-shell" design. Each half of the oven contains an electric radiant heating element surrounded by a ceramic insulation block. Air or nitrogen gas is introduced through a purge ring with the gas flow directed over the heated coils providing heat transfer by convection. For subambient testing, both gas and liquid nitrogen are fed through the purge ring. Liquid nitrogen usage is kept to a minimum by switching from gas to liquid nitrogen only when cooling is required. Temperature Sensors are used both in the oven and below the lower plates for temperature measurement and control. The ETC also features a camera viewer option installed through the chamber's right-side ceramic block. The camera option features multiple light sources, remote focusing, and can be used over the entire temperature range. Used in conjunction with the streaming video and image capture software, real-time images can be displayed in the software and an image is stored with each data point for subsequent viewing. The ETC camera viewer is an ideal tool for data validation.



ETC Geometry Accessory Kits

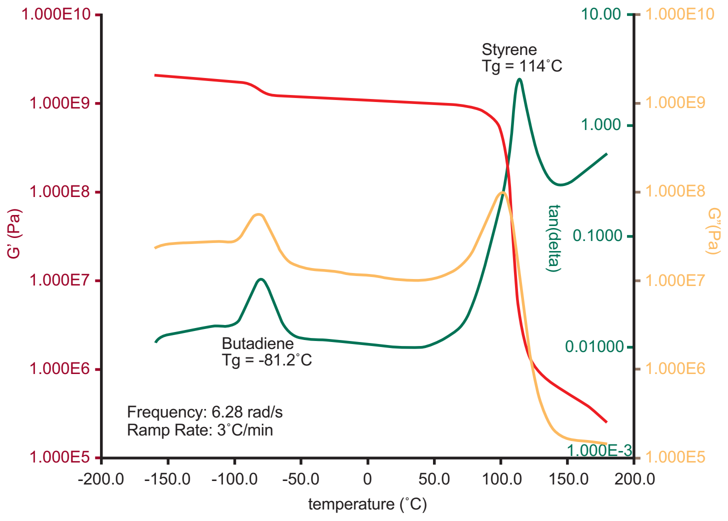
The ETC features five standard geometry accessory kits configured for thermoplastics and rubber, thermosetting and other curing systems, solid polymers, pressure sensitive adhesives, and asphalt binder. In addition, a wide variety of stainless steel geometries of various diameters and cone angles, and a wide variety of disposable plates, are available to fully accessorize the temperature system. The AR-G2 features patented Smart Swap™ geometries that are automatically recognized and configured for use.

- Parallel Plate Kit for most common samples
- Disposable Plates for reactive systems
- Torsion rectangular for solid samples
- Pressure sensitive adhesives kit
- Asphalt Binder Kit with 8 and 25 mm diameter plates and molds

Solid Polymer Rheology Using Torsion Rectangular Clamps

The ETC oven has the ability to characterize the viscoelastic properties of rectangular solid samples up to 5 mm thick, 13 mm wide, and 50 mm long. The figure shows an example of an oscillatory temperature scan on a solid ABS specimen run at 3°C/min from –160 to 200°C. The ABS is a copolymer that exhibits two glass transition temperatures: one at –81°C associated with the Butadiene and one at 114°C associated with Styrene. Transitions or relaxations of molecular segments are observed as step changes in the storage modulus (G′), and as peaks in the loss modulus (G″) and damping (tanδ). The magnitude and shape of these parameters will depend on chemical composition as well as physical characteristics such as crystallinity, orientation, fillers, and degree of cross-linking.

ABS Oscillation Temperature Ramp in Torsion Rectangular

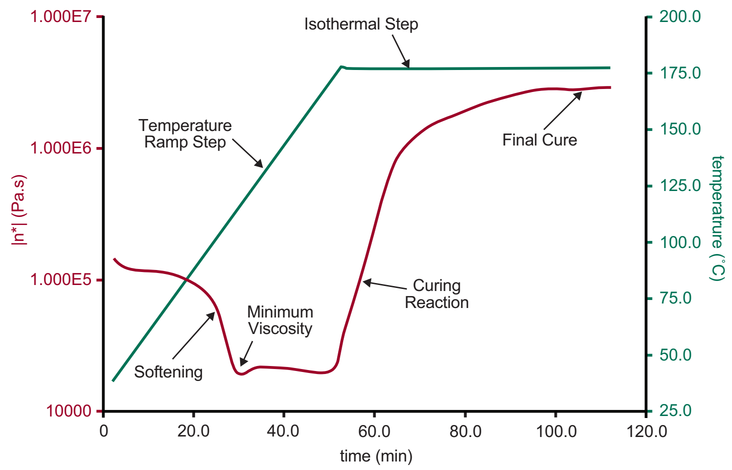


EHP and ETC High Temperature Polymer Application

Themoset Cure Using Disposable Plate Kit

Changes in the viscoelastic properties of reactive systems provide valuable information about their processing and end use properties. Often these materials can start in the form of a low viscosity liquid, a paste, or even a powder, and after reacting end as a high modulus solid. If the material hardens and adheres to the testing surface, low cost disposable parallel platens are required for testing. The figure to the right shows the complex viscosity from a typical oscillatory temperature ramp and hold curing test conducted on a B-stage prepreg using 25 mm diameter disposable plates. The resin is impregnated on a woven glass matrix, which becomes part of the permanent high strength composite structure once the resin is cured. Upon heating the resin softens until reaching a minimum viscosity, which is a very important processing parameter. If it is too high or too low, it will not flow and coat the matrix uniformly, leaving voids, creating flaws in the composite. Eventually the viscosity starts building and the curing reaction takes off dramatically. The temperature is held constant at the processing temperature and the viscosity is monitored until the viscosity reaches a plateau indicating the completion of the reaction.

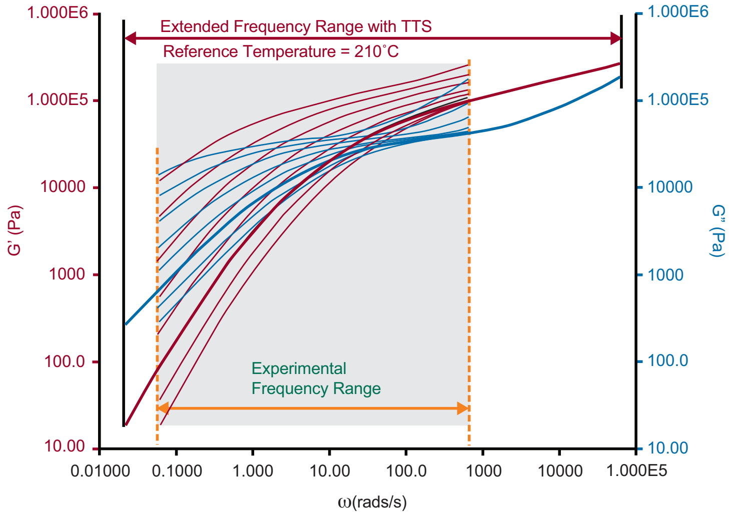
B-Stage Prepreg Temperature Ramp and Hold Cure



Themoplastic Polymer Rheology Using Parallel Plate

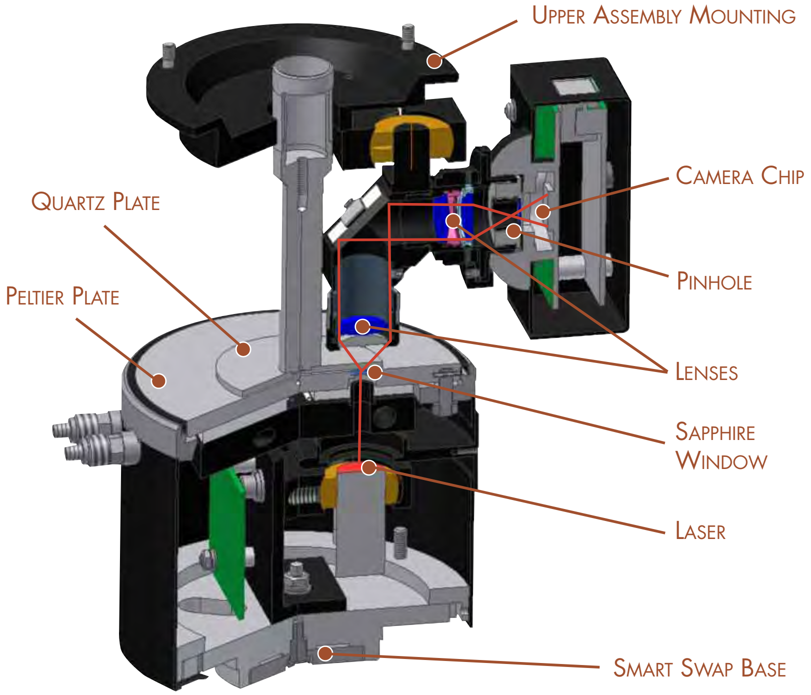
Parallel Plate geometries are most popular for testing of thermoplastic polymer melts. An example of polystyrene characterized over a temperature range of 160°C to 220°C is shown in the figure to the right. Frequency sweeps were run at multiple temperatures over an experimental range of 0.06 to 628 rad/s. The magnitude of the viscoelastic properties, storage modulus (G′) and loss modulus (G″), for the individual sweeps can be seen to decrease with increasing temperature over this frequency range. Since polymer melts are viscoelastic, their mechanical response will be time dependent, so low frequency corresponds to long time behavior. Time Temperature Superposition (TTS) is used to widen the range of data to higher and lower frequencies and generate a mastercurve at a reference temperature pertinent to the application. The polymer's molecular structure dictates the magnitude and shape of the G′ and G″ curves.

Polystyrene Frequency Sweeps from 160°C to 220°C



Small Angle Light Scattering (SALS) Accessory

The AR Series Small Angle Light Scattering System, AR-SALS, is an option for simultaneously obtaining rheological and structural information, such as particle size, shape, orientation and spatial distribution. The AR-SALS is available for the AR-G2 and AR 2000ex Rheometers. The option incorporates TA Instruments' Smart Swap™ technology bringing a new level of speed and simplicity for making simultaneous rheology and SALS measurements. The system can be installed, aligned, and ready for measurements in as little as five minutes. It features patented Peltier Plate temperature control[1] and the scattering angle (θ) range over which measurements can be made is ~ 6° to 26.8°. The scattering vector range (q) is 1.38 μm^{-1} to 6.11 μm^{-1} and the length scale range is about 1.0 μm to ~ 4.6 μm.



Technology

The SALS accessory consists of upper and lower assemblies and quartz plate geometry. The lower assembly includes an integrated Class 2 laser with 0.95 mW diode and wavelength λ = 635 nm situated below a patented[1] Peltier Plate with a 5 mm diameter quartz window. The Peltier Plate surface is stainless steel with a temperature range of 5 to 95°C. The upper assembly consists of a set of lenses and a camera. The scattered light is focused through a lens pair mounted within a height-adjustable cap to focus at varying sample depth. The light is then focused through a second lens and sent through an adjustable polarizer for both polarized and depolarized measurements. Finally, the scattering is collected through a pinhole and recorded by the Camera. The upper geometry is a 50 mm diameter, 2 mm thick optical quartz disk. To comply with the single-point correction for the parallel plates, the laser is set at 0.76 times the plate radius which is 19 mm from the axis of rotation of the plate. This arrangement keeps the SALS system compact, while allowing for quick and reproducible positioning and focusing. A set of neutral density filters is available as an option to reduce laser intensity.

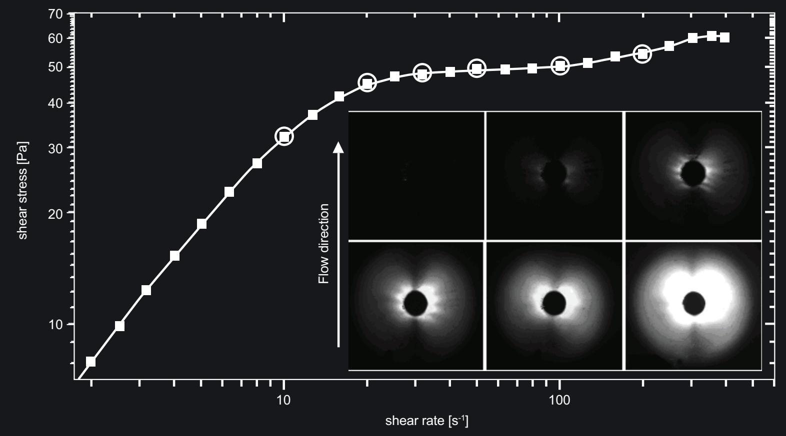
(1) Patent # 7,500,385

Features and Benefits

- q vector range ~1.38 μm^{-1} to 6.11 μm^{-1}
- Objects length scale range ~ 1 to 4.6 μm
- Scattering angle ~ 6° to 26.8°
- Wavelength 635 nm
- Compact upper assembly requiring minimal adjustment
- Smart Swap™ lower assembly with factory aligned laser
- Class 2 laser - No safety issues
- Adjustable laser intensity with optional neutral density filters
- Variable depth focus to adjust for different geometry gaps
- Adjustable polarizer for scattering in parallel or perpendicular to the incident light
- Image focused directly onto camera chip – Does not require screen or darkened room
- Quantitative measurements possible by calibration with monodisperse Polystryrene beads.
- Optional Analysis Software
- Patented Peltier Plate temperature control

Shear-Induced Phase Separation of Micellar Solutions:

Self-assembled surfactant micelles show a variety of shear-induced microstructural transformations that are important for material formulation and function for a wide array of applications. Simultaneous measurements of rheology and surfactant microstructure, using SALS, under shear provide a valuable tool in examining shear-induced transitions in such fluids. The data in the figure show scattering images captured synchronously with rheometry data on a surfactant system. At low shear rates below the stress plateau, no measurable scattering is obtained from the sample, suggesting no large-scale structuring of the fluid. However, in the stress plateau, a strong anisotropic scattering pattern develops with increasing shear rate, which exhibits "butterfly" patterns that result from the scattering associated with phase separation, where interface between the two phases contributes to the strong scattering.

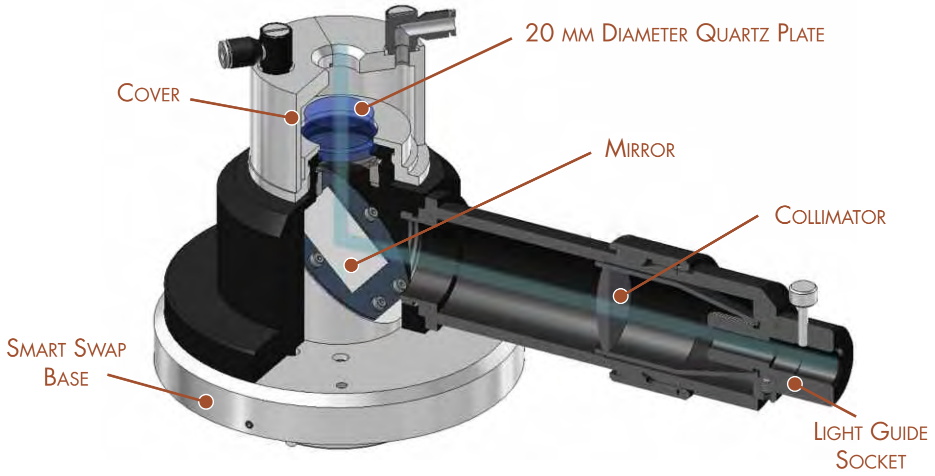


UV Curing Accessories

UV-curable materials are widely used for coatings, adhesives, and inks. When these materials are exposed to UV radiation, a fast cross-linking reaction occurs, typically within less than a second to a few minutes. Two Smart Swap™ accessories for rheological characterization of these materials are available for the AR-G2 and AR 2000ex rheometers. Onc accessory uses a light guide and reflecting mirror assembly to transfer UV radiation from a high-pressure mercury light source. The second accessory uses self-contained light emitting diodes, LED, arrays to deliver light to the sample. Accessories include 20 mm quartz plate, UV light shield, and nitrogen purge cover. Optional temperature control to a maximum of 150°C is available using AR Series Electrically Heated Plates (EHP) option. Disposable plates are available for hard UV coatings which cannot be removed from the plates once cured.

UV Light Guide Accessory Technology

The AR UV Light Guide accessory includes a lower Smart Swap assembly with quartz plate, light source mount, collimator, 5 mm diameter light guide, and UV mercury lamp source (Exfo Omnicure S2000). It provides a broad wavelength spectrum from 250 nm to 600 nm, with a primary peak at 365 nm. The maximum output intensity is greater than 300 mW/cm². External filter holder and filters are available for the light source.

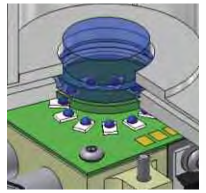


Features and Benefits

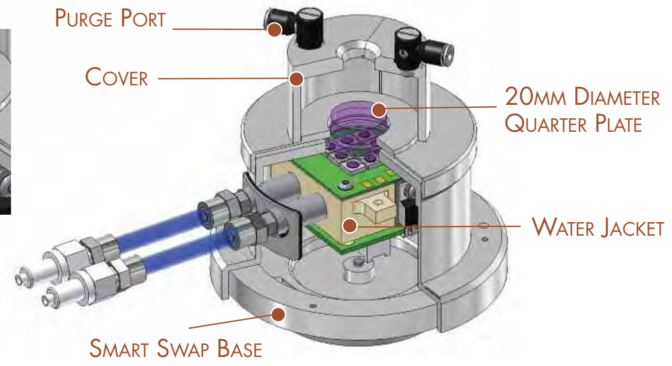
- Convenient compact Smart Swap™ option
- Collimated light and mirror assembly insure uniform irradiance across plate diameter
- Maximum intensity at plate 300 mW/cm²
- Broad range spectrum with main peak at 365 nm
- One system with specific wavelengths accessible through filtering options
- Cover with nitrogen purge ports
- Optional disposable acrylic plates
- Optional temperature control to 150°C
- Software programable trigger time and intensity

UV LED Accessory Technology

The UV LED accessories use arrays of light emitting diodes that provide single peak wavelength light sources. The LED array is mounted on a PCB and is fixed to a water jacket that cools the LED's during use. Like the UV light guide system, LED's are pre-aligned to insure uniform irradiance across the surface of the plate. There are two LED accessories available at wavelengths of 365 nm and 455 nm. The maximum output UV intensity is 150 mW/cm² and 350 mW/cm², respectively. They are fully integrated with the rheometer through Smart Swap option. Trigger time and intensity are conveniently programmed through the software.



Blue Light LED Array
Wavelength = 455 nm



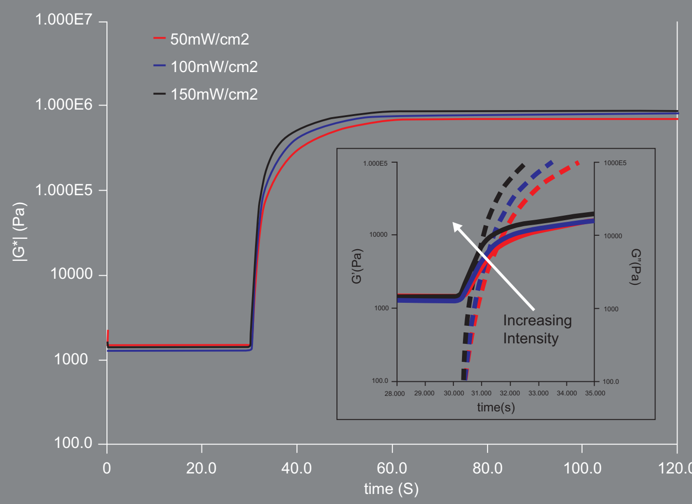
Features and Benefits

- New technology replaces mercury bulb systems
- 365 nm wavelength with peak intensity of 150 mW/cm²
- 455 nm wavelength with peak intensity of 350 mW/cm²
- No intensity degradation over time
- Even intensity across plate diameter, LED positioned directly below plate
- Compact and fully integrated design including power, intensity settings and trigger
- Cover with Nitrogen purge ports
- Optional disposable Acrylic plates
- Optional temperature control to 150°C

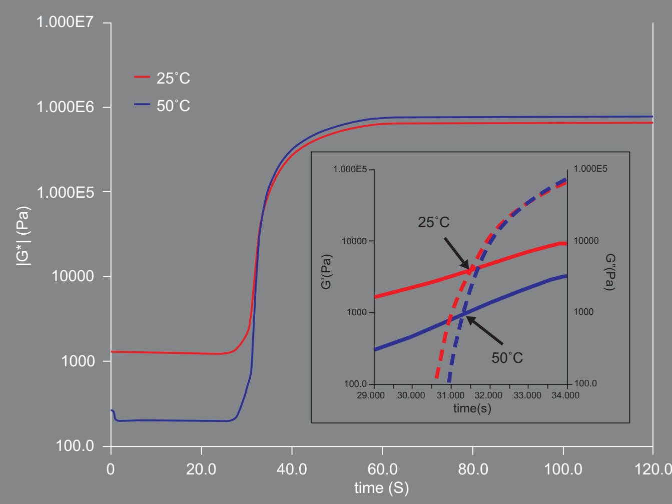
UV Option Application

These accessories allow the study of UV Reactions by monitoring the elastic (G') and viscous (G") moduli. Examples 1 and 2 show results of a pressure sensitive adhesive, PSA, characterized on the LED 365 nm option and the Light Guide. In Example 1, the PSA was held at an isothermal temperature of 25°C and the intensity was varied between 50 to 150 mW/cm². In Example 2, experiments were run at intensity level of 100 mW/cm², at two temperatures of 25°C and 50°C. In both examples, the sample is measured for 30 sec before the light is turned on. The data show faster reaction kinetics with both increasing intensity (Example 1) and increasing temperature (Example 2), as evidenced by the shorter time for crossover of G' and G". The curing reaction happens in less than two seconds. The fast data acquisition of the AR rheometers, (up to 50 pts/sec), clearly resolves the liquid to solid transition. Note that changing the intensity and temperature by small amounts shifts the crossover point by a fraction of a second. This information is important for understanding adhesive control parameters for high-speed UV curing processes, as well as for understanding differences in initiators when formulating materials.

Example 1: Effect of Intensity/Light Guide



Example 2: Effect of Temperature/LED

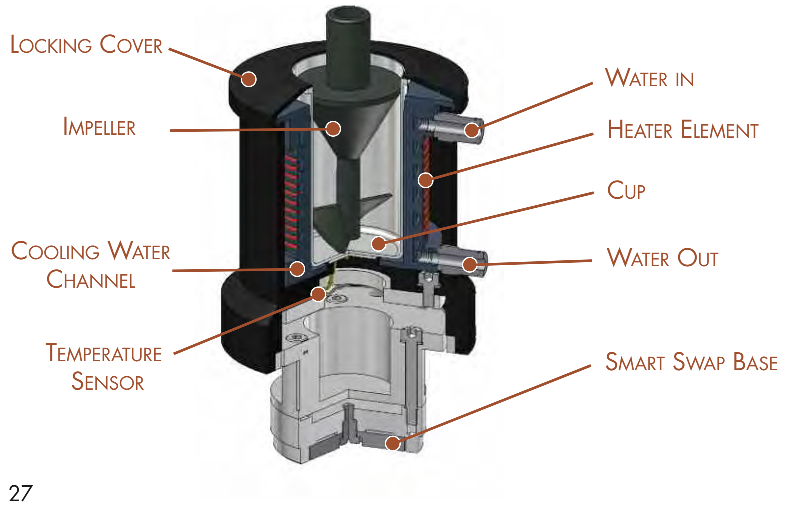


Starch Pasting Cell

The Starch Pasting Cell (SPC) is a Smart Swap™ accessory available on all AR Rheometers. The option provides a more accurate and powerful tool to characterize the gelatinization of raw and modified starch products as well as the properties of the starch gels. It can also be used for characterizing many other highly unstable materials. It uses an innovative impeller design for mixing, reduction of water loss, and control of sedimentation during testing. The actual sample temperature is measured and controlled in a temperature chamber with heating/cooling rates up to 30°C/min.

Technology

The Starch Pasting Cell consists of the cell jacket, an impeller, and aluminum cup with locking cover. The cell jacket houses a heating coil and liquid cooling channel which surround the Aluminum cup for fast heating and cooling. A Platinum Resistance Thermocouple (PRT) is located in intimate contact with the bottom of the cup for precise and accurate sample temperature control. The impeller is designed with blades at the bottom for sample mixing. Solvent loss is minimized via a conical ring at the top of the rotor, which acts to condense water or other solvents that vaporize during heating, and return it to the bulk sample.



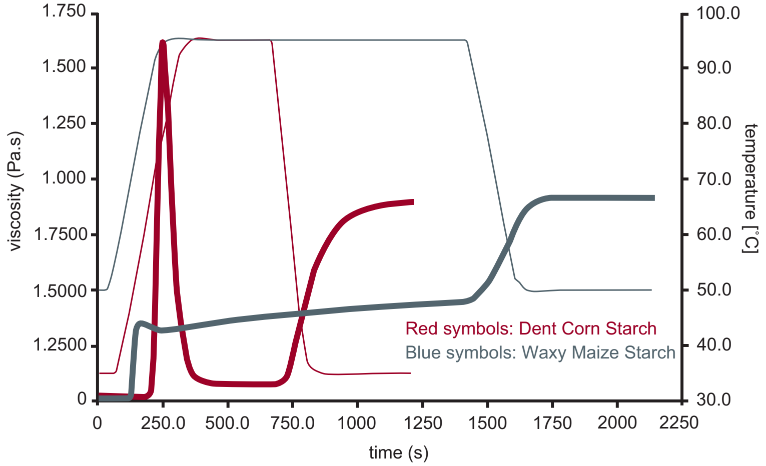
Features and Benefits

- Smart Swap temperature system
- Heating/Cooling rates up 30°C/min
- Higher accuracy for greater reproducibility
- Robust Cup and Impeller
- Impeller keeps unstable particles suspended in liquid phase during measurements
- Impeller design minimizes loss of water or other solvents
- Sample temperature measured directly
- All rheometer test modes available for advanced measurements on gelled starches and other materials
- Optional conical rotor for traditional rheological measurements

Gelatinization of Starch Products

Starch is not only a food product; functionally modified starches are widely used in the industry including adhesives, paper, coatings, wood, packaging, pharmaceutical, and many others. When starch is heated above a critical temperature, the starch granules undergo an irreversible process, known as gelatinization. The properties of the starch gels depend on the origin of the raw starch (crop, potatoes, etc.), the environmental conditions (seasons) or the modification. The viscosity curve, referred to as pasting curve, produced by heating and cooling starches generally has a similar characteristic shape. The figure to the right shows two scans each of both a Dent Corn and Waxy Maize starch. The benefit of the AR Rheometer starch cell design can easily be observed in the unprecedented reproducibility of the pasting curves for theses two starch products.

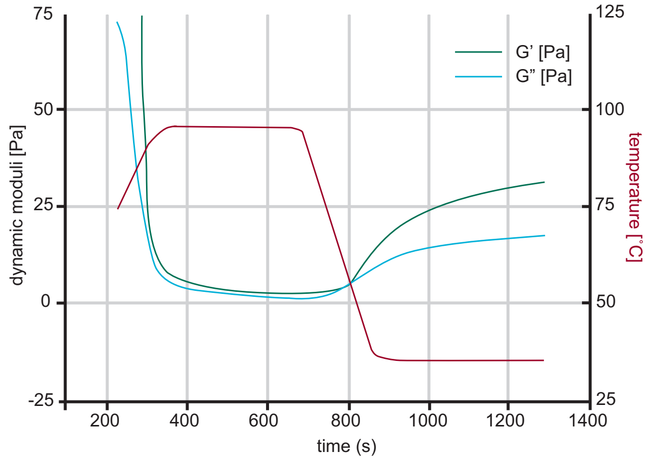
Two Scans each of Dent Corn and Waxy Maize Starch



Advanced Starch Rheology

In addition to measuring the characteristic pasting curve of starch products, the starch cell brings new testing capabilities for measuring properties of the starch gels. The figure to the right shows additional data obtained on Dent Corn starch using an oscillation test to monitor the gelation process of the starch under negligible shearing. In this test, the sample is sheared while ramping temperature to keep starch particles suspended. At 75°C, when the viscosity is high enough to inhibit particle settling, the steady shear was stopped and testing was continued at a small oscillating stress. The figure shows storage modulus, G′, and the loss modulus, G″. which provide extremely sensitive information about the structural characteristics of the starch gelation and final gel. This enables the development of valuable structure-property relationships. The ability to make these sensitive measurements is not possible on traditional starch characterization instrumentation.

Dent Corn Starch Gelatinization



NOTES

Local Offices

New Castle, DE USA	+1-302-427-4000
Lindon, UT USA	+1-801-763-1500
Hialeah, FL USA	+1-305-828-4700
Crawley, United Kingdom	+44-1293-658900
Shanghai, China	+86-21-64956999
Taipei, Taiwan	+88-62-25638880
Tokyo, Japan	+81-3-5759-8500
Seoul, Korea	+82-2-3415-1500
Bangalore, India	+91-80-2319-4177-79
Paris, France	+33-1-30-48-94-60
Eschborn, Germany	+49-6196-400-600
Brussels, Belgium	+32-2-706-0080
Etten-Leur, Netherlands	+31-76-508-7270
Sollentuna, Sweden	+46-8-555-11-521
Milano, Italy	+39-02-265-0983
Barcelona, Spain	+34-93-600-93-32
Melbourne, Australia	+61-3-9553-0813
Mexico City, Mexico	+52-55-52-00-18-60