# RUBBER TESTING

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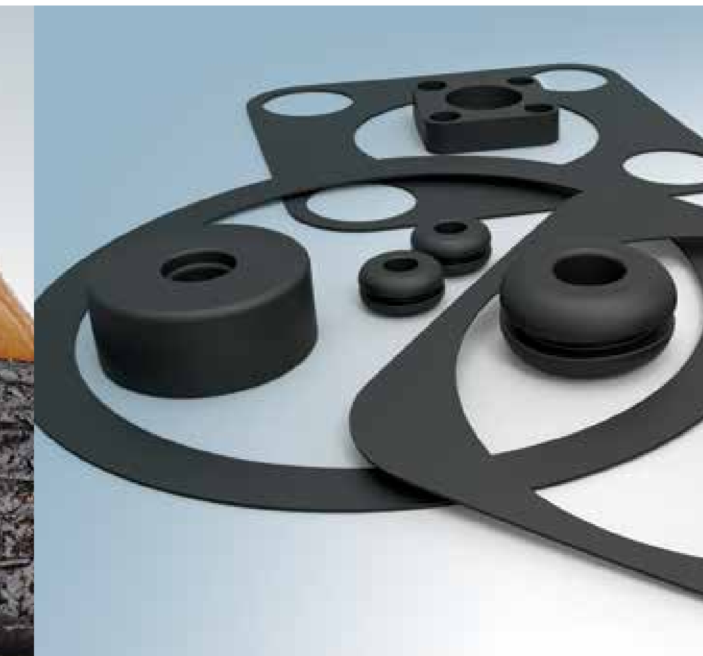
## Rubber Testing



# Rubber Testing

TA Instruments introduces a complete line of new instruments for the measurement of rheological and physical properties of polymers, rubber and rubber compounds at all stages of manufacture. The new rubber testing instruments include a Rubber Process Analyzer (RPA), Moving Die Rheometer (MDR), Mooney Viscometer, Automated Density Tester and Automated Hardness Tester.

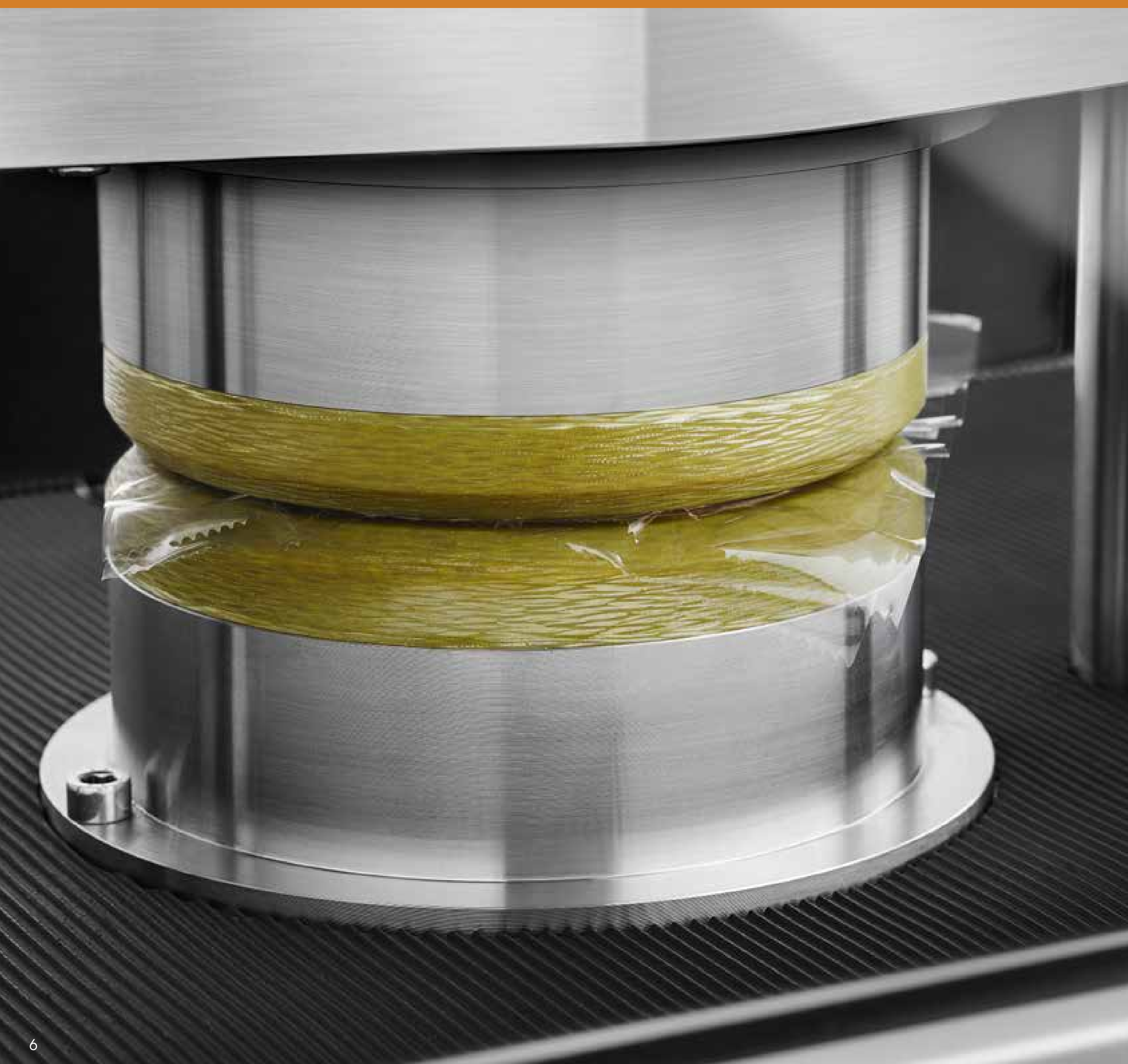
All TA Instruments rubber testing systems are manufactured to exacting mechanical standards and with the latest measurement technology for the most accurate, reliable, and reproducible data available. Available automation systems allow for maximum unattended laboratory productivity in all test environments. Relevant ASTM, DIN, and ISO standards are easily met, as are demands for advanced testing, making these instruments the ideal choice for quality control, analytical, and research needs.

As the world leader in viscoelastic measurements for over forty years, TA Instruments brings technical expertise in making the most accurate physical property measurements and provides a world-renowned global support network.

# RPA *flex*

RUBBER PROCESS ANALYZER

The RPA *flex* provides the highest quality of rubber rheometer data in a user-configurable platform, making it the perfect fit for labs that need more testing capability than an MDR alone can provide. The RPA *flex* includes all the same high quality features of the RPA *elite*, including ultra-rigid test frame, variable direct drive motor, proprietary high-stiffness torque transducer, precision temperature control with optional cooling, available Autosampler, and powerful Scarabaeus Software for control and analysis. The base level model includes a wide continuous strain range and fixed frequency enabling all MDR standard cure testing (ASTM/DIN/ISO). In addition, the RPA *flex* can be configured to include, or be upgraded to include, any or all of the features of the RPA *elite* including variable strain, frequency, stress relaxation, advanced oscillation, LAOS testing, and sample pressure measurement.



## Features

- High resolution variable direct drive motor for absolute strain control
- Proprietary high-stiffness, wide torque range transducer with high sensitivity for noise-free data
- Extremely rigid test frame for accurate compliance-free data
- Upgradeable to the widest range of tests and testing conditions
- Fully programmable variable testing parameters (strain, frequency, temperature, and closing pressure)
- Available autosampler for unattended operation
- Pneumatic locking cylinders with adjustable platen closing force and pressure sensor
- User calibration and user-replaceable seals


## Specifications

| | |
|---|---|
| Frequency | Fixed: 1.67 Hz (100 cpm)<br>Optional: 0.001 to 50 Hz (0.167E-3 to 3,000 cpm) |
| Amplitude | Standard: ±0.1° to ±7° arc (continuous)<br>Optional: ± 0.005 to 360° arc (continuous) |
| Strain | Standard: ±1.4 % to ±100 %<br>Optional: ±0.07 % to ±5000 % |
| Torque | 0.01 to 25 N.m |
| Temperature | Ambient to 230 °C<br>18 °C to 230 °C with Enhanced Cooling Option |
| Maximum Ramp Rate | 80°C/min (1.33°C/s) |
| Die Type | Sealed bicone, 0.48 mm gap |
| Sample Volume | 4.5 $cm^3$ |
| Platen Sealing Pressure | Fixed: 4.5 bar Optional: 1 to 8 bar Adjustable |
| Sample Pressure | Optional: 0 to 8,000 kPa |
| Test Modes | **Standard method**<br>Curemeter/Vulcanization both isothermal and temperature ramp<br>**Optional methods**<br>Strain (sweep, offset, LAOS), Frequency Sweep,<br>Stress Relaxation, Advanced (multi-frequency, abitrary waveforms) |
| Measured Data | Torque, Temperature, Frequency, Amplitude, Sample Pressure (optional) |
| Calculated Data | **Standard:** Process parameters including: ts1, TC10, TC50, TC90, S'min, S'max, scorch time, cure rate, and viscoelastic properties including: G', G", G*, S', S", S*, tan δ, η', η", η*<br>**Optional:** LAOS Lissajous curves, non-linearities in stress and strain |
| Standards | **Standard:** ASTM D5289, ISO 6502, DIN 53529<br>**Optional:** ASTM D6048, D6204, D6601, D7050, D7605; ISO 13145 |

# technology

TA Instruments is the world's leading supplier of analytical instrumentation for the measurement of viscosity and viscoelastic properties. The new rubber testing instruments are uniquely designed to deliver the highest quality of torque, amplitude, frequency, temperature, and pressure measurement and control. TA Instruments is the **CLEAR CHOICE** for testing rubber products at all stages of manufacture.

## Rigid Testing Platform

All TA Instruments rubber rheometers and viscometers are built with an ultra-stiff testing frame and crosshead which eliminates the ***effects of instrument compliance*** on test data. Instrument compliance, or instrument deformation, produces erroneously low values of measured properties such as modulus and torque, irregular strain and torque waveform signals, and other errors.

Large diameter steel rods and a thick crosshead brace in the H-shaped load frame provide unmatched rigidity to resist instrument deflection while the motor deforms the sample. This special design ensures that the commanded strain is achieved with each cycle of deformation, even for highly filled, fully cured rubbers. Additionally, a non-compliant system allows for truly sinusoidal strain profiles under all conditions. This can be verified by continuous Fourier Transform analysis of the deformation and measured torque signals which is available in the Scarabaeus Software. The superior design also guarantees smooth travel, proper alignment, and precise application of vertical load.



## Direct Drive Motor

Powerful direct drive motors apply precise deformation in all TA Instruments rubber rheometers and viscometers. A high quality rheological or dynamic measurement relies on the precise application of a constant rate, step, or periodic deformation. In a direct drive system the start-up delays, compliance, and translational losses seen in clutch or belt-driven configurations are eliminated. The superior TA Instruments motor design ensures that the most accurate and repeatable deformations are always applied to the sample.

The RPA *elite* and RPA *flex* provide continuously variable strain and frequency ranges for flexible testing. The RPA *elite* applies the **highest combination of frequency and amplitude** in any rubber rheometer. This provides important material information such as:

- The linear viscoelastic response of highly filled rubbers at low strains
- Behavior at extreme processing and use conditions characterized by high strains
- Terminal material behavior exhibited at low frequencies
- Response to high speed deformations measured at high frequencies

## High-Stiffness Torque Transducer

The RPA *elite* and RPA *flex* benefit from a proprietary wide range ultra-stiff torque transducer. This rugged, non-compliant device measures the widest range of torques accurately and precisely. This greatly improves the accuracy and precision of measured torque, modulus, and viscosity values.

## Advanced Data Processing

The complex deformations and stress-strain response common to rubber testing demand the most advanced data processing techniques. TA Instruments rubber rheometers utilize a state-of-the-art 20-bit encoder and advanced data sampling technique to perform calculations based on a Fast Fourier Transform (FFT) analysis using 90 data points for each cycle of oscillation.

The RPA *elite* and RPA *flex* are capable of measuring and reporting non-linearities in torque and displacement. Higher harmonics that indicate non-linearity in the applied displacement or measured torque are reported for each data point, alerting the operator with a simple indicator if test conditions are not ideal and storing this information for subsequent data validation.

Crosshead Bearing

## High Pressure Pneumatic System

All TA Instruments rotorless rheometers and curemeters employ a high pressure pneumatic system to **seal the sample properly and reproducibly.** The high capacity pneumatic system applies up to 8 bar nominal pressure to the sample during gap closure. Proper alignment and the use of mechanical bearings ensure efficient transfer of load from the system to the sample without load frame losses. Actual sealing pressure is measured directly and recorded. This high pressure automated sample containment removes operator dependence and tightly contains the test specimen. This sealing process is particularly important for materials that undergo positive or negative volumetric changes with curing and highly stiff materials such as carbon-filled fluoroelastomers.

## Cooling Options

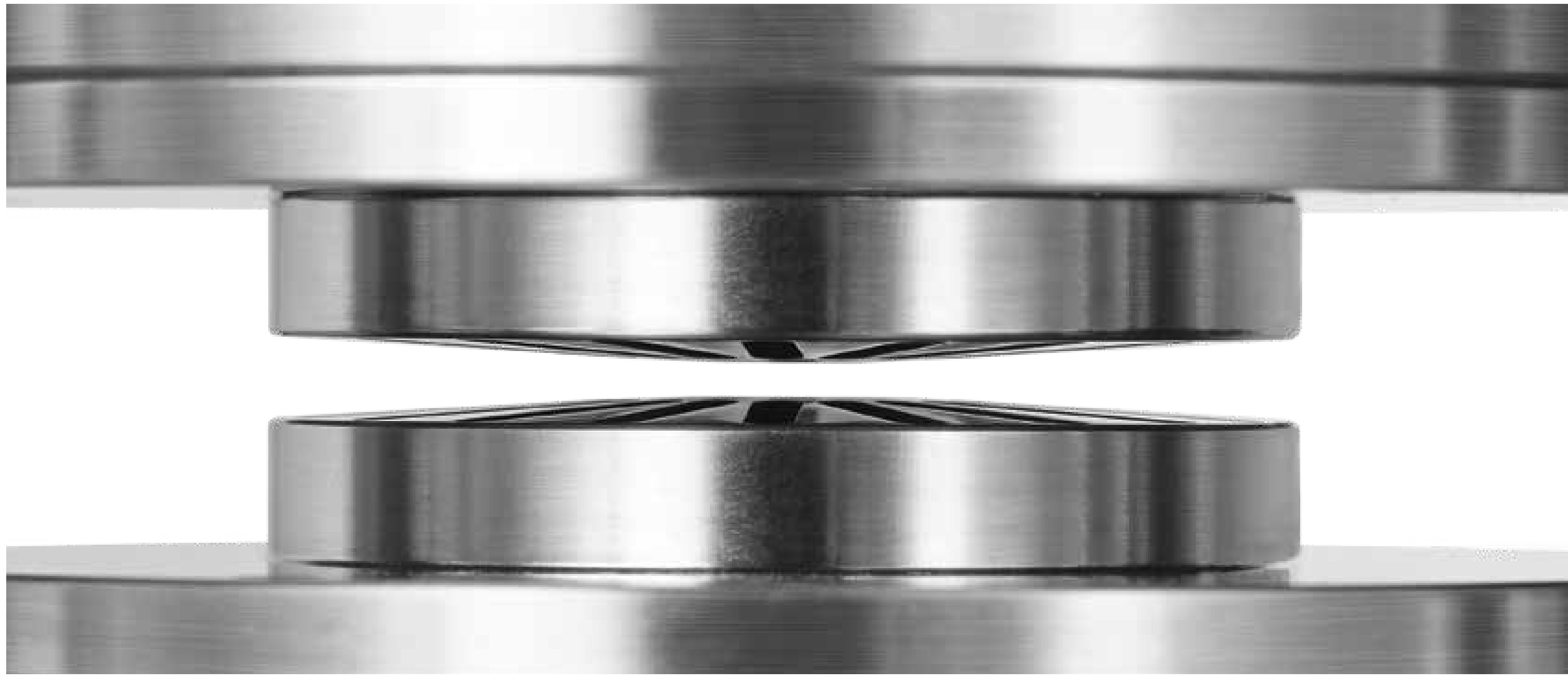
All rotorless rheometers are compatible with either the Air Cooling System or Enhanced Cooling System. The **Air Cooling System** uses ambient air to expedite temperature changes above ambient conditions and improve temperature stability near room temperature.

The **Enhanced Cooling System** is a mechanical device that employs pressurized air to cool the test environment, allowing characterization of rubbers at temperatures as low as 18 °C and greatly accelerating cooling time between non-isothermal experiments. The system has no moving parts, making it extremely reliable and easy to use.

## Testing Dies and Rotors

The MDR *one*, RPA *flex*, and RPA *elite* rotorless shear rheometers employ the industry-standard sealed cavity biconical die design. The dies are constructed from durable, high-stiffness, low thermal expansion stainless steel to **minimize system compliance and prevent gap changes** with temperature. The test fixtures are connected directly in line with the motor below for precision deformation control and the torque transducer above for accurate measurement.

Direct contact electric heaters mounted within the dies provide exceptional temperature control and stability under isothermal, step and temperature ramp conditions. This highly responsive system returns rapidly to the programmed test temperature upon the addition of a cold sample, providing the most representative values for scorch time and other cure characteristics. Extremely durable user-replaceable seals provide absolute sample containment at all temperatures and conditions.



Die Surfaces

Torque Calibration Standard

## Rotorless Rheometer

RPA and MDR die surfaces feature an optimized arrangement of radial serrations to guarantee constant sample contact at even the highest strain values. Polyester or polyamide films may be used to facilitate sample release and avoid the need to clean dies between experiments.

Torque calibration is made simple with a certified torque calibration standard. This allows the user to calibrate the instrument directly, increasing data confidence and operation time, and reducing the reliance on service engineers for calibration.

## Mooney Viscometer

The MV *one* Mooney Viscometer includes both large (38.1 mm) and small (30.48 mm) diameter rotors. Both rotors are endorsed by international standards and can be selected for measurement of low or high viscosity rubbers or polymers. Both rotor types can be used in conjunction with polyester or polyamide films to simplify instrument cleaning and reduce time between runs. Rotors are designed with low mass to optimize thermal response and transient speed changes at the beginning and end of shearing steps.

Calibration is software-driven and does not require the use of external weights, fixtures, or reference materials. A weight of known mass is connected by a well-defined radius, creating a constant torque value. The software-driven torque calibration routine uses this internal standard to ensure utmost data accuracy.

Mooney Viscometer Rotors

Integrated Torque Calibration





## Sample Preparation

Sample preparation for RPA, MDR, and Mooney instruments is made safe and simple with the VS Volumetric Sample Cutter. This dual-action pneumatic system allows for the preparation of uncured rubber specimens of a user-defined volume.

Preparing samples in this well-controlled fashion **reduces operational variability, greatly improving overall experimental precision**.

The VS comes standard with a closing pressure of 6 bar. The sample is first compressed to the user-prescribed thickness, then cut to the die diameter. The closing pressure, and sample volume are user-adjustable. An optional booster is available to increase operating pressure to 8 bar for highly filled and stiff materials. Two-handed operation and lateral guards guarantee safe operation at all times.

## Sample Cutting Dies

Sample dies for either the Mooney Viscometer or rotorless rheometers and curemeters can be removed and exchanged quickly and easily. The MV *one* Mooney Viscometer uses two 40 mm diameter samples, one above and one below the rotor. A hole is punched in the center of the sample to ease insertion. Standard RPA and MDR samples are single piece discs cut to meet volume specifications per ASTM, DIN, and ISO standards.

# reliable automation

## RPA AND MDR

All TA Instruments rotorless curemeters and rheometers are compatible with the highly reliable rubber automation system. This carousel-based autosampler allows for the unattended testing of rubber samples. Coupled with the Scarabaeus Software automated analysis, statistics, and control chart generation, the automated rheometer becomes a highly integrated part of the manufacturing control process. This improved data throughput is also invaluable for screening multiple formulations in a research or product development environment.

Samples are moved from the autosampler tray to the test position using a suction transfer system. This system is highly tolerant of non-ideal sample geometries, so uncured rubber from many sources can be used. Samples are loaded on a carousel and more specimens may be added while another test is in progress, allowing for uninterrupted productivity.

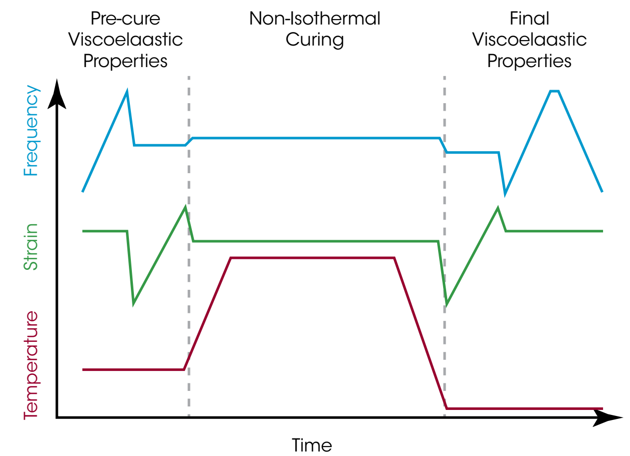
An automated film transfer system removes the previously tested specimen and advances the film roll to prepare for the next experiment. An integrated sensor alerts the operator at the end of the roll so it can be replaced in a timely manner.

# scarabaeus software

The Scarabaeus Software for instrument control and data analysis is a powerful and versatile system for programming experiments, providing quick feedback of results, and managing data from all rubber testing instruments. The Scarabaeus Software was developed with customers from the rubber industry and is designed to meet the specific need of production and research.

## Simple Instrument Control, Flexible Programming

Instrument control software is preloaded with test programs for the most common experiment types, enabling simple operation by new users. Multi-step tests can be easily programmed to collect many types of data from a single specimen, or to mimic an industrial curing or other processing sequence.

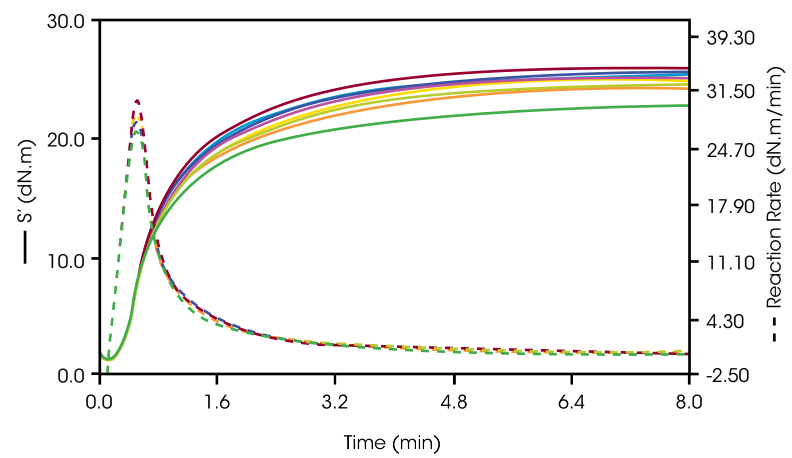


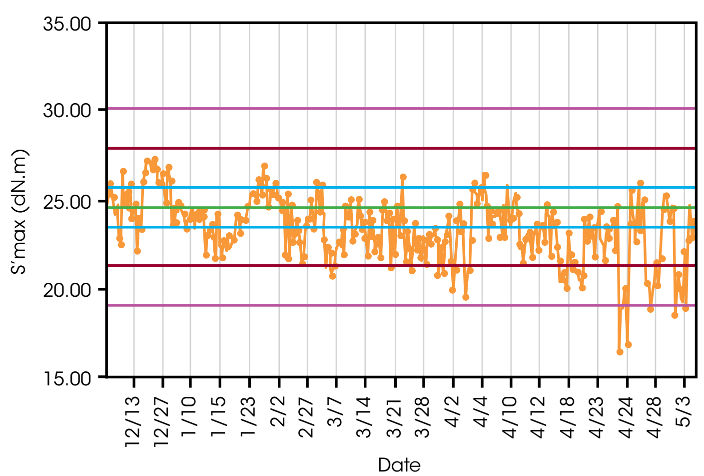

## Quick Operator Feedback

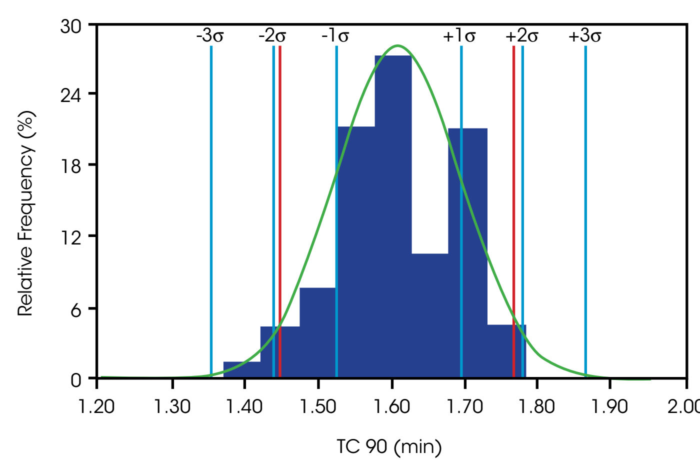
Qualification of multiple lots of similar materials is made easy with quick operator feedback. Predefined test parameters with tolerances can be assigned for a given material. Upon completion of a test, a simple pass/fail indicator shows whether the specimen falls within the acceptable limits for the selected material, allowing meaningful decisions to be made quickly and easily.

## Statistical Process Control

Test data is readily converted into actionable information for process control and manufacturing. Automated data analysis can be programmed based on typical performance metrics, such as minimum and maximum torque, scorch times, conversion times, and more. These data are compared against user-defined limits and are used to track processes using histograms, control charts, and summary reports.



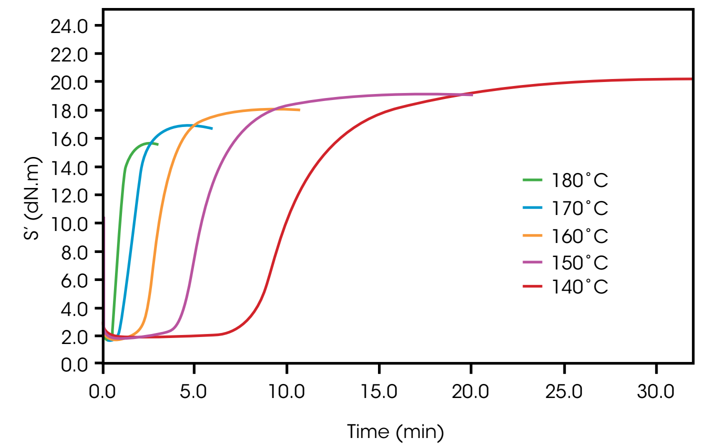



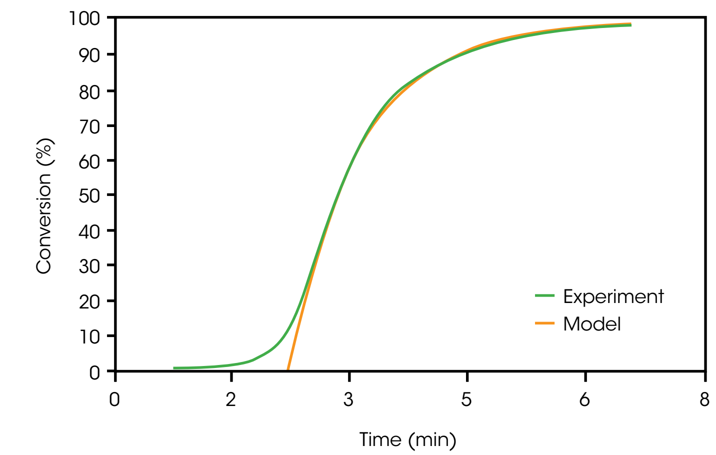



## Advanced Data Analysis and Modeling: Curing Kinetics

Isothermal curing data at multiple temperatures can be analyzed according to a rubber-specific methodology to determine curing kinetics parameters. This modeling system can determine:

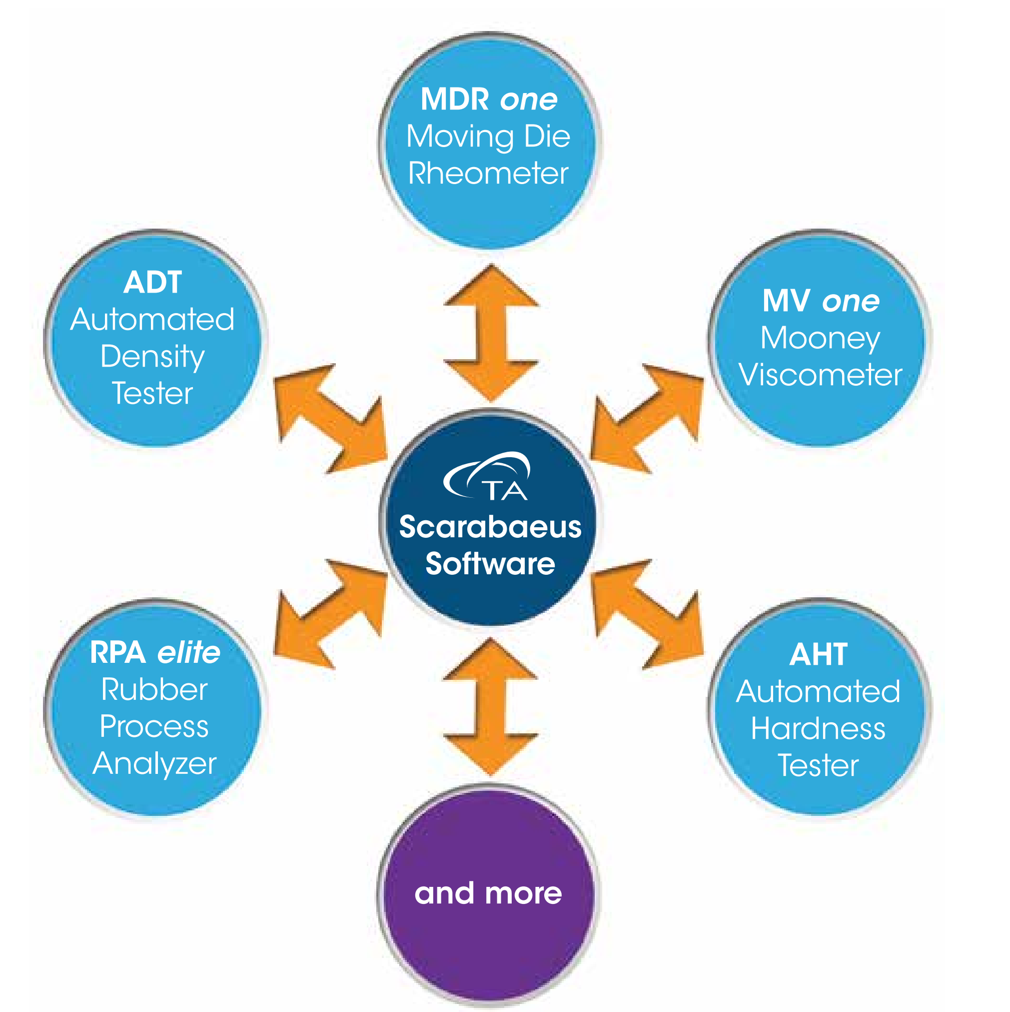
- Reaction Rate
- Reaction Order, *n*
- Rate Constant, *k*
- Incubation Time, $t_i$
- Arrhenius Activation Energy, $E_a$




## Designed for Integration

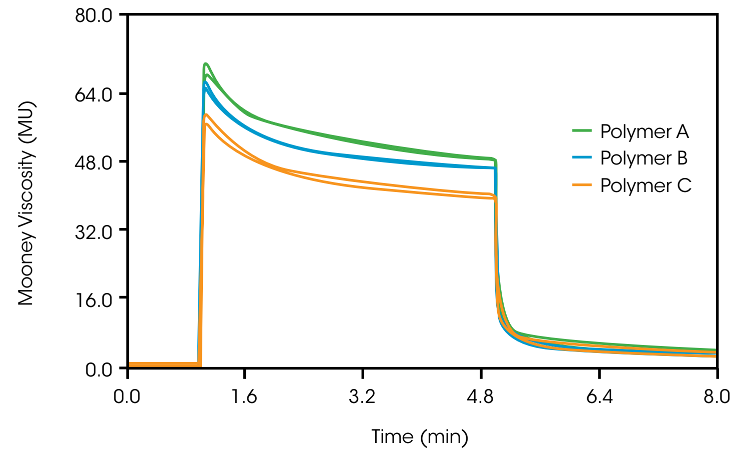
The Scarabaeus Software system for instrument control and analysis integrates and organizes data from multiple instruments and historical tests. Data from RPA, MDR, Mooney Viscometer, Hardness, and Density tests can be organized, compared, and analyzed by material type, inventory order, date, and more. Advanced integration with even greater capability is also available.



# applications

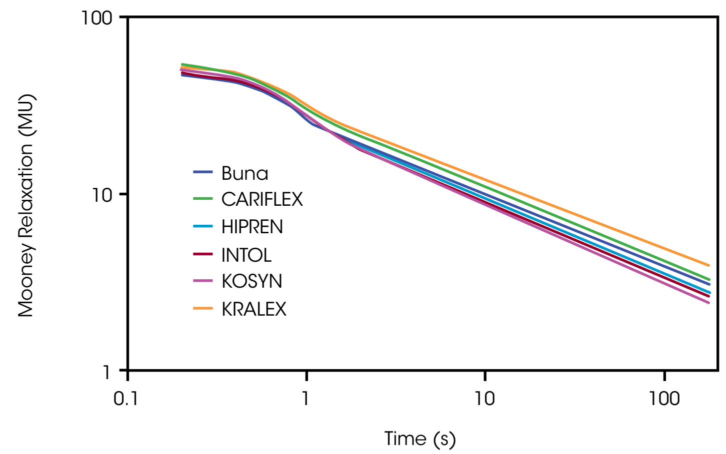
## Mooney Viscosity

The Mooney Viscosity test is a well-established method for characterizing uncured rubber materials. Following well-defined standard procedures, the sample is preheated for a defined period, then sheared at a constant rate. The Mooney Viscosity is recorded from the end of this deformation stage. In the present example, the outstanding precision of the MV *one* Mooney Viscometer is demonstrated. Three polymer samples were tested in duplicate. The outstanding run-to-run reproducibility and the ease of distinguishing one polymer from another is clear.



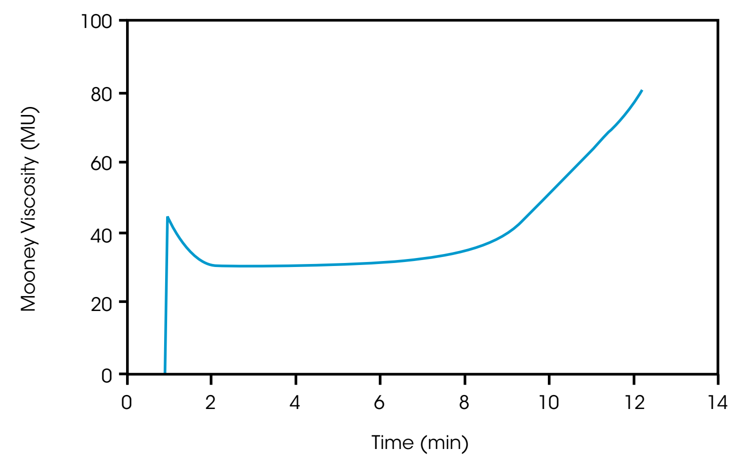

## Mooney Stress Relaxation

While the Mooney Viscosity experiment is typically indicative of polymer viscosity, stress relaxation can be used to identify elasticity. Upon completion of the Mooney Viscosity measurement, the rotor is stopped immediately and the torque decay is observed. The slope of this decay is indicative of polymer elasticity, which may be related to a branched architecture and correlates well with extrudate swell in rubber processing.

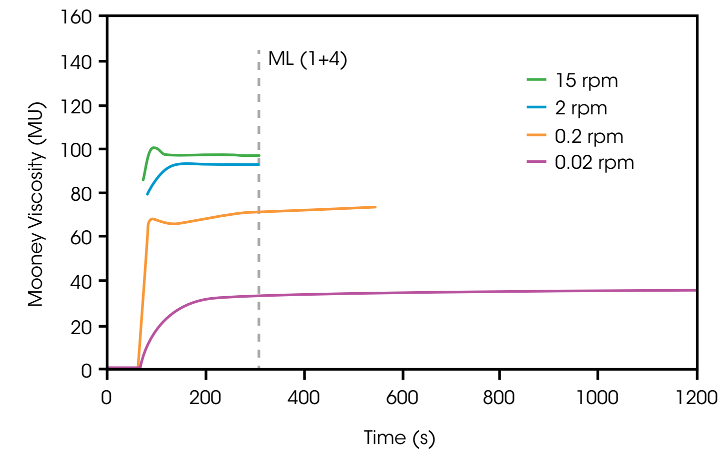


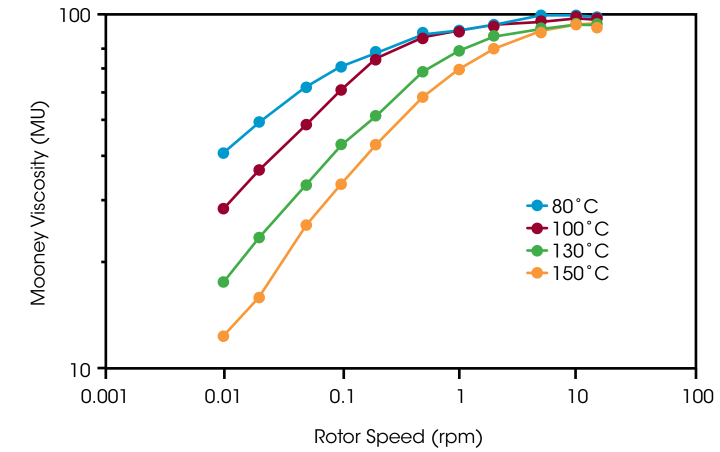

## Mooney Scorch

The Mooney Viscometer can also be used to measure the initial rate of vulcanization. In this example, a styrene butadiene rubber (SBR) was tested for prevulcanization characteristics at 150°C using the small rotor. For this simple experiment the initial Mooney viscosity, minimum viscosity, scorch times, and cure index are the most commonly reported values.



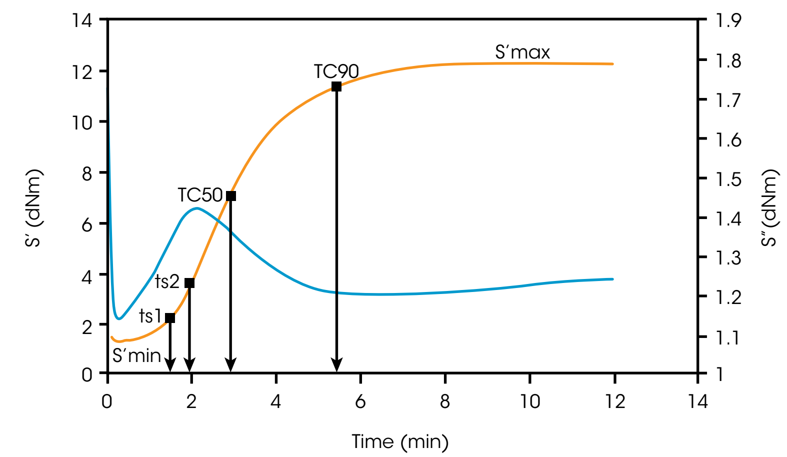


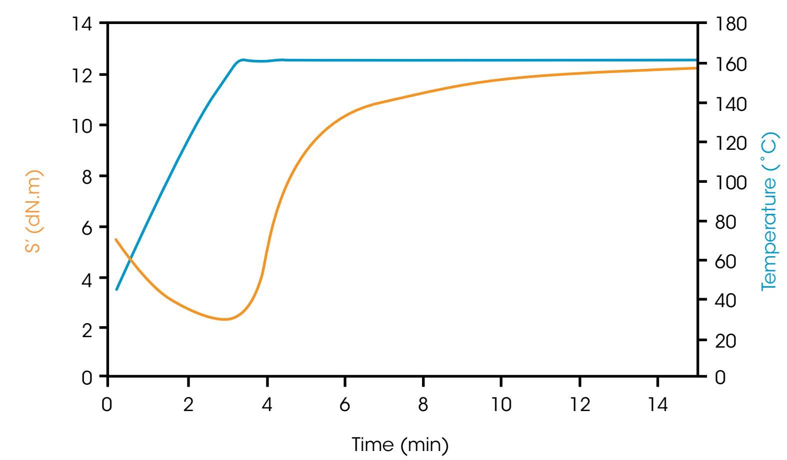

## Mooney Viscosity at Multiple Rates

In addition to the viscosity at a single rate and temperature, the MV *one* Mooney Viscometer can measure viscosity at a range of shear rates and temperatures. This range of rates allows a more complete understanding of the polymer behavior, especially a tendency for shear thinning. Low rates in Mooney Viscosity experiments can also be beneficial for measuring highly elastic materials that are otherwise inaccessible to Mooney Viscosity measurements.




## Isothermal Cure

Isothermal cure experiments are critical for rubber and elastomer processing. The TA Instruments rubber rheometers provide high precision data that is simple to analyze. All the important characteristics, such as minimum and maximum viscosity, scorch time, and conversion time can be calculated easily and automatically. The data can also be handled in its complete graphical form for comparison or alternative analyses.

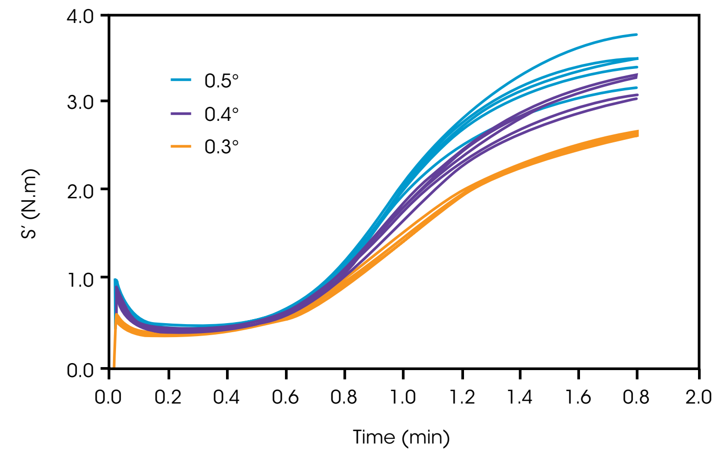


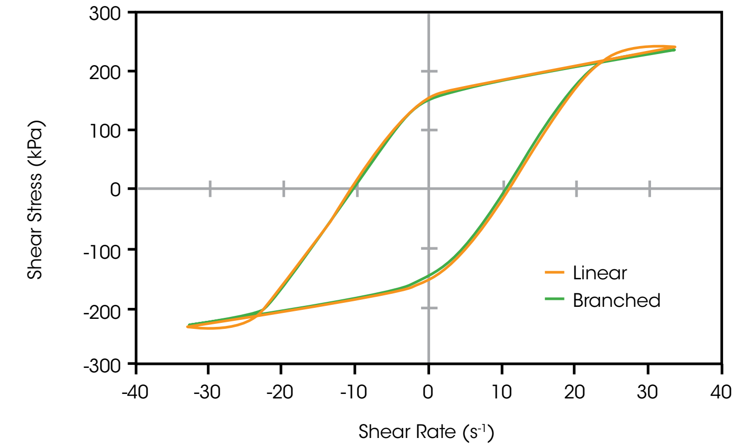

## Non-isothermal Cure

In addition to the industry-standard isothermal cure methods, the RPA and MDR can perform non-isothermal cure experiments. These experiments can be programmed to follow virtually any temperature profile and are especially valuable when simulating manufacturing processes that are not isothermal. Non-isothermal curing experiments may also be coupled with isothermal tests such as strain and frequency sweeps before or after cure to provide a more complete material data set before, through, and after cure.

## Isothermal Curing at Variable Strain

While standard test methods often call for a single strain and frequency value to be used for all materials (0.5°, 1.67 Hz), these are not always the ideal conditions for every material. In the present example, the sample material is tested by isothermal cure at three deformation amplitudes, five times each. At the standard of 0.5° and 0.4° the experimental variability is extremely broad. This is because these experiments are performed at strains beyond the linear viscoelastic limit for this material. Testing at a smaller amplitude (0.3°) produces valid data with greatly improved reproducibility.



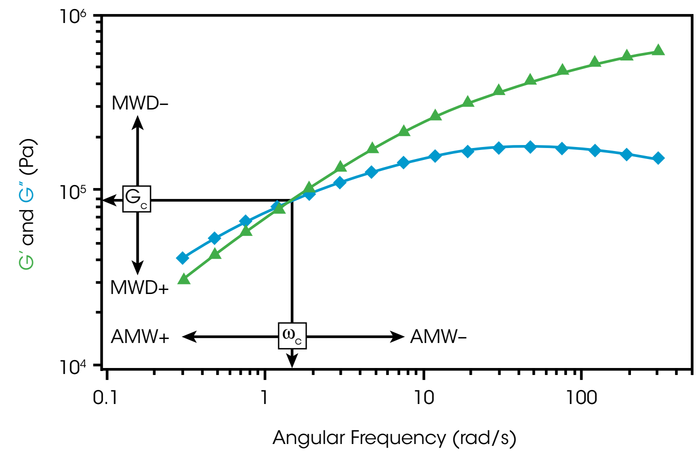



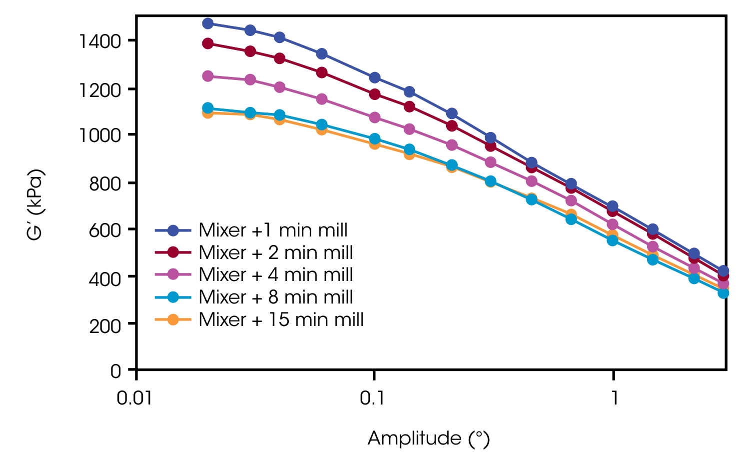

## High Strain Non-Linear Behavior

The viscoelastic response of a branched material at very high strains differs not only in magnitude from its linear counterpart, but also in type. The careful examination of a polymer's stress-strain response at high strains reveals features associated with filler content and structure, as well as polymer architecture. In the present example, qualitatively different features are observed at large strains for two EPDM materials: a linear polymer and a branched polymer. Both exhibit identical Mooney viscosities, but markedly different high strain behavior. Both the uncorrelated data, and the FT analysis of periodic data are available through the Scarabaeus Software to allow in-depth analysis of this new type of data.

## Isothermal Frequency Sweep

Measuring the frequency-dependent viscoelastic properties of a material is a powerful way to understand its molecular structure. A frequency sweep as shown can reveal information about the average molecular weight (crossover frequency) and molecular weight distribution (crossover modulus).



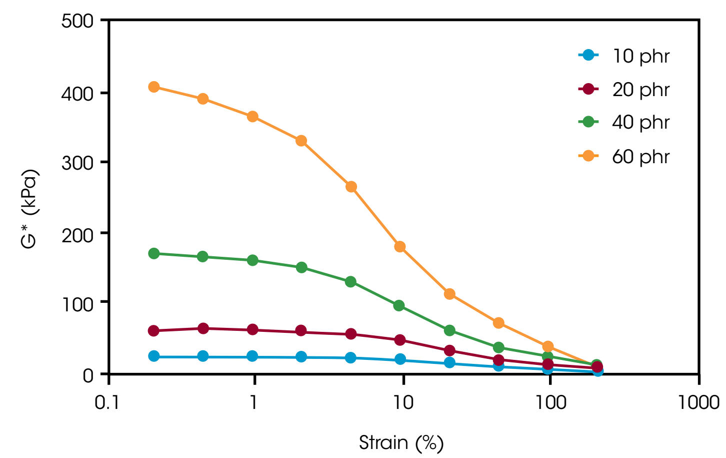



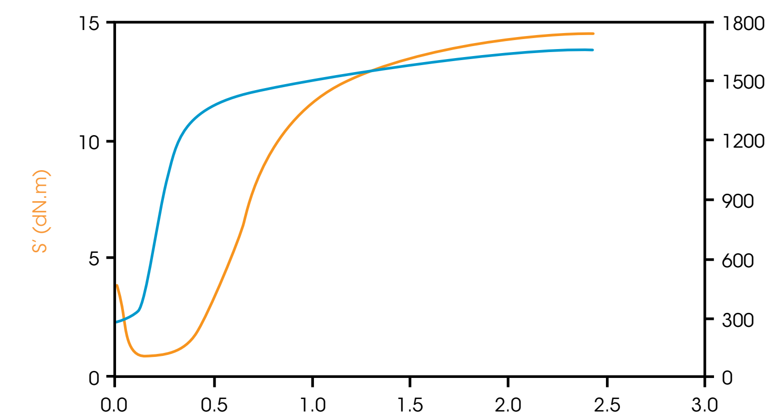

## Time Dependent Structure Breakdown

The van der Waals interactions that lead to increased modulus in carbon black filled rubber are highly sensitive to processing. In this example, identical samples are subjected to different lengths of milling after being removed from the mixer. Carbon network structure is reduced with each increase in milling time up to 8 minutes, after which the modulus was unchanged with increased milling. This provides critical information about the amount of milling needed to create a consistent workable material.

## Strain Sweep for Filler Loading

The strain-dependent modulus is particularly important as an indicator of the amount and type of rubber filler dispersion and interaction. In the present example, the impact of carbon black addition at five different levels is seen in the low strain region. High strain behavior is generally insensitive to filler addition, as it is less sensitive to filler-filler interaction and more dependent on polymer molecular weight, and polymer-filler interactions.




## Cure with Blowing Reaction

Final product density and mechanical performance are often enhanced through the use of blowing agents to create a cellular architecture. These blowing agents generate gas during decomposition in parallel with the curing reaction. Monitoring sample pressure through the curing reaction is an effective way to quantify the blowing reaction, allowing for the characterization of curing and blowing in a single experiment. These two processes must be balanced in order to form the desired cell architecture in the finished product.

# specifications

## Feature Summary

| | MDR *one* | RPA *flex* | RPA *elite* |
|---|---|---|---|
| Torque Transducer | High-Stiffness | Proprietary Ultra-High-Stiffness | Proprietary Ultra-High-Stiffness |
| Torque Range | 0.01 to 25 N.m | 0.01 to 25 N.m | 0.1 to 20 N.m |
| Motor | Direct Drive | Direct Drive | Direct Drive |
| Strain | | | |
| 0.2°, 0.5°, 1.0°, 3.0° | ● | ● | ● |
| 0.1° to 7° (continuous) | — | ● | ● |
| 0.005° to 360° (continuous), Strain Sweep, Offset, LAOS | — | ○ | ● |
| Frequency | | | |
| 1.67 Hz (100 cpm) | ● | ● | ● |
| 0.001 Hz to 50 Hz, Frequency Sweep | — | ○ | ● |
| Sealing Pressure | | | |
| Fixed: 4.5 bar | ● | ● | ● |
| Variable: 1 to 8 bar | — | ○ | ● |
| Sample Pressure Measurement | — | ○ | ○ |
| Air Cooling System (ambient to 230°C) | ○ | ○ | ● |
| Enhanced Cooling System (18°C to 230°C) | ○ | ○ | ○ |
| Isothermal and Non-Isothermal Curing | ● | ● | ● |
| Non-Linearity Measurements | — | ○ | ● |
| Stress Relaxation | — | ○ | ● |
| Advanced Oscillation (arbitrary wave, multi-frequency) | — | ○ | ● |
| Autosampler | ○ | ○ | ○ |

● Included

○ Optional

— Not Available

# partner for success
ADVANCED RUBBER CHARACTERIZATION

- Initial Installation & Training
- Local Service
- TechTip Videos
- Theory and Applications Courses
- eTraining
- Hands-On Training Courses

## Global Support

TA Instruments has a reputation for product innovation and quality. Combined with our dedication to support, it's easy to see why TA Instruments enjoys outstanding customer loyalty. Service is a cornerstone of the support we provide our customers every day. The vast array of training products ensures that our customers utilize their instruments effectively, maximizing return on investment. To provide this support, TA Instruments has assembled the largest worldwide team of service and support professionals in the industry. With direct support staff in 23 countries and 5 continents, TA Instruments can extend its exception support to you, wherever you are.

Others promise good support. Talk to our customers and learn how TA Instruments consistently delivers exceptional support.

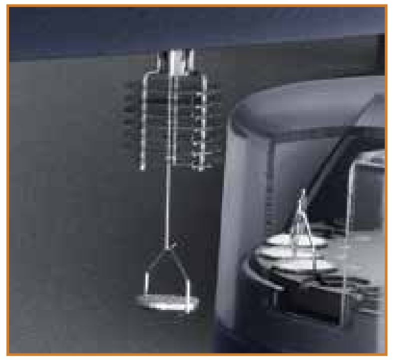
## Focus, Innovation

TA Instruments is committed to designing, manufacturing, and delivering high value analytical instrumentation based around a few core measurements. We strive for the most accurate and precise measurement of temperature, mass, displacement, and force. These four components form the foundation of a wide array of analytical techniques.

### DSC

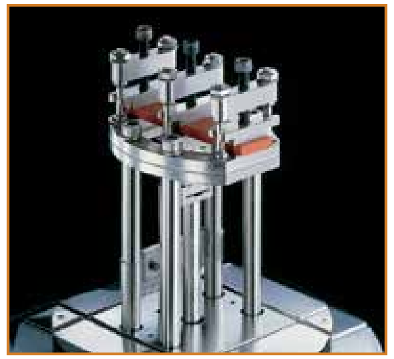
- Curing profiles and kinetics
- Residual cure
- Phase transitions
- Oxidation Induction Time

### TGA



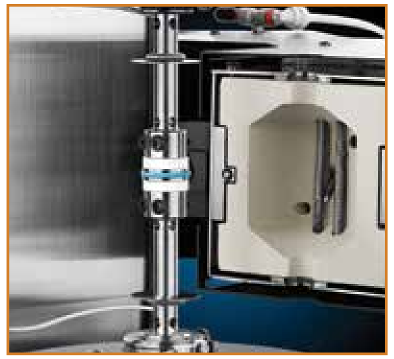
- Compositional analysis
- Thermal Stability
- Evolved Gas Analysis by Mass Spec or FTIR
- Decomposition kinetics

### DMA



- Final viscoelastic properties
- Finished part analysis
- Phase Transitions
- Filler effects

### Dielectric

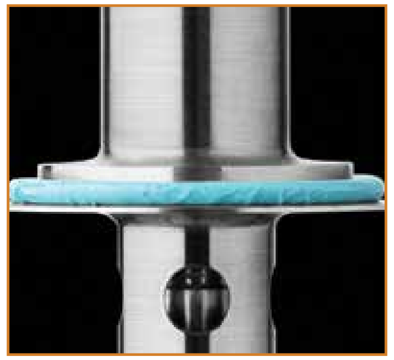


- Dieletric Properties
- Filler networks
- High frequency relaxation
- Phase transitions
- Compatible with DMA or rheometer systems

### Thermal Conductivity

- Highly accurate, simple measurement
- Heat dissipation
- Cure process modeling

### Rheometers



- High sensitivity rheology
- Solutions, polymers, coatings
- -160 ˚C to 600 ˚C
- Many sample types
- Complementary accessories

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