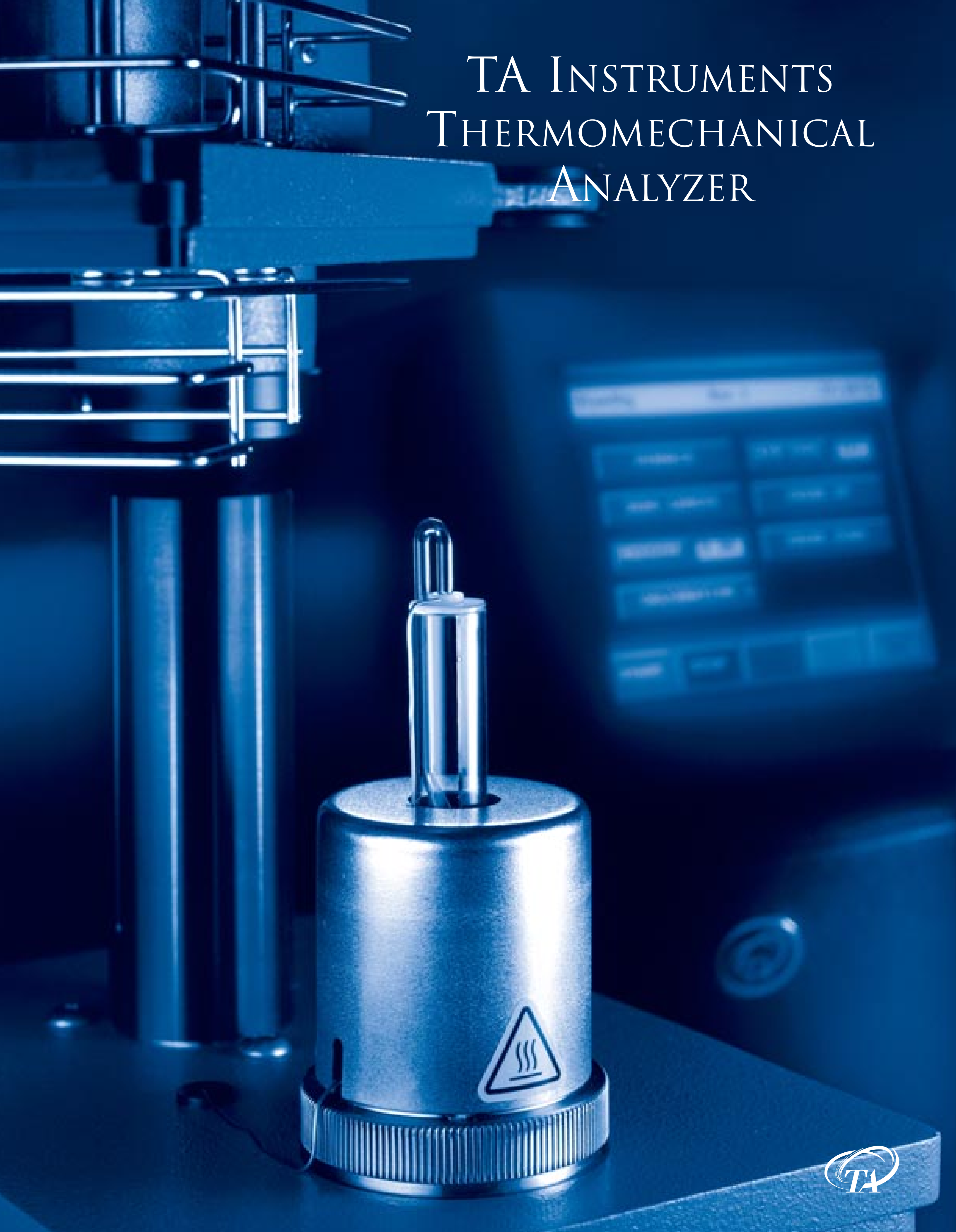
TA Instruments
Thermomechanical
Analyzer



TA INSTRUMENTS Q SERIES™
THERMOMECHANICAL ANALYZER

The Q400 is a sixth-generation product from the world leader in thermal analysis. Its performance, ease-of-use, and reliability aptly demonstrate our long experience in thermomechanical analysis design.

The Q400EM is a high-performance, research-grade thermomechanical analyzer (TMA), with unmatched flexibility in operating modes, test probes, fixtures, and available signals. For standard TMA applications, the Q400 delivers the same performance and reliability. It is ideal for research, teaching, and quality control applications, with performance equivalent to competitive research models.

TECHNICAL SPECIFICATIONS

	TMA Q400EM	TMA Q400
Temperature Range (max)	-150 to 1,000°C	-150 to 1,000°C
Temperature Precision	+ /- 1°C	+ /- 1°C
Furnace Cool Down Time (air cooling)	<10 min from 600°C to 50°C	<10 min from 600°C to 50°C
Maximum Sample Size - solid	26 mm (L) x 10 mm (D)	26 mm (L) x 10 mm (D)
Maximum Sample Size - film/fiber	26 mm (L) x 0.5 mm (T) x 4.7 mm (W)	26 mm (L) x 0.5 mm (T) x 4.7 mm (W)
Measurement Precision	+/- 0.1 %	+/- 0.1 %
Sensitivity	15 nm	15 nm
Dynamic Baseline Drift	<1 µm (-100 to 500°C)	<1 µm (-100 to 500°C)
Force Range	0.001 to 1 N	0.001 to 1 N
Force Resolution	0.001 N	0.001 N
Frequency	0.01 to 2 Hz	Not Available
Mass Flow Control	Optional	Optional
Atmosphere (static or controlled flow)	Inert, Oxidizing, or Reactive Gases	Inert, Oxidizing, or Reactive Gases

Operational Modes		
Standard	Included	Included
Stress/Strain	Included	Not Available
Creep	Included	Not Available
Stress Relaxation	Included	Not Available
Dynamic TMA (DTMA)	Included	Not Available
Modulated TMA™ (MTMA™)	Included	Not Available

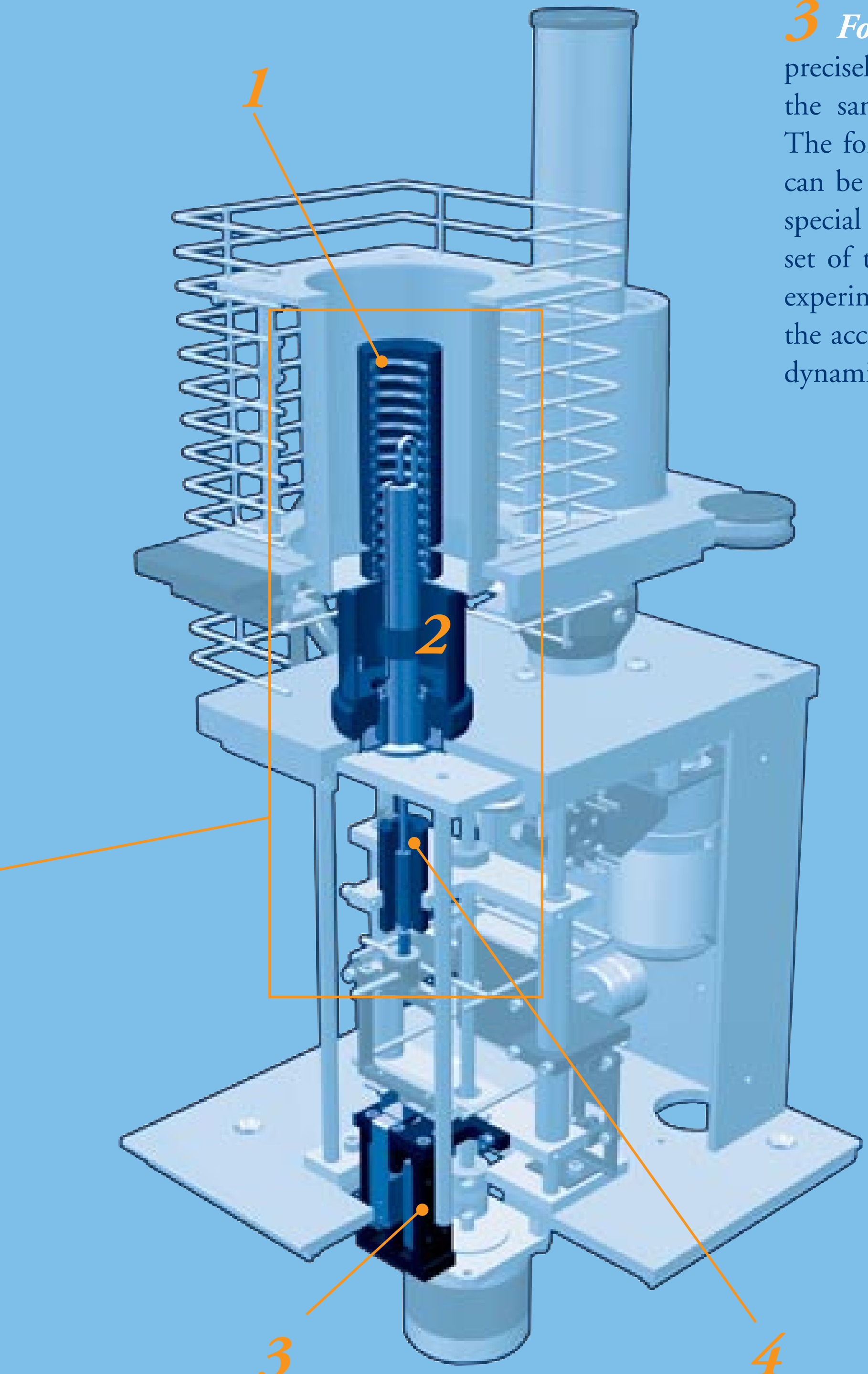
Note: The Q400 can be field upgraded to the Q400EM.

DESIGN FEATURES AND BENEFITS

A thermomechanical analyzer measures sample dimensional changes under conditions of controlled temperature, time, force, and atmosphere. Our engineering experience in design and integration of critical furnace, temperature and dimension measurement, and atmosphere control components meld with powerful, flexible, software to optimize the many tests that the Q Series TMA can perform.

1 Furnace The Q400 features a rugged and reliable furnace. Its customized electronics provide excellent heating rate control and rapid response over a wide temperature range. Furnace raising and lowering is software controlled. **Benefits:** The design ensures long life and performance consistency. The excellent heating rate control provides for superior baseline stability and improved sensitivity, while the rapid response permits Modulated TMA™ operation. Furnace movement provides operational convenience, and easy access to the sample chamber.

2 Sample Chamber Located in the furnace core, the easily accessed chamber provides complete temperature and atmosphere control for sample analysis. Purge gas regulation is provided by an optional digital mass flow controller. **Benefits:** These include enhanced data quality, ease-of-use, and productivity. The open design simplifies installation of available probes (see Modes of Deformation), sample mounting, and thermocouple placement. Data precision is enhanced by mass flow control of the purge gas.



3 *Force Motor* A non-contact motor provides a precisely controlled, friction-free, calibrated force to the sample via the measurement probe or fixture. The force is programmable from 0.001 to 1 N, and can be increased to 2 N by addition of weights to a special tray. A precision sine wave generator provides a set of ten individual frequencies for use in dynamic experiments. **Benefits:** The motor smoothly generates the accurate and precise static, ramped, or oscillatory dynamic force necessary for quality measurements in all modes of operation. The choice of frequencies allows optimization of dynamic TMA (DTMA) experiments in compression, 3-point bending, or tension modes of deformation.

4 *Linear Variable Differential Transducer* The heart of the Q400 TMA sample measurement system is the precision, moveable-core, linear variable differential transducer (LVDT). **Benefits:** It generates an accurate output signal that is directly proportional to a sample dimension change. Its precise and reliable response over a wide temperature range (–150 to 1,000°C) makes for reproducible TMA results. Its location below the furnace protects it from unwanted temperature effects and ensures stable baseline performance.

MODES OF DEFORMATION

The Q400 offers all the major TMA deformation modes necessary to characterize solids, foams, films, and fibers. These include compression, tension, and 3-point bending.

COMPRESSION

In this mode, the sample is subjected to either a static, linear ramp, or dynamic oscillatory force, while under a defined temperature program, and atmosphere. Sample displacement (strain) is recorded by either expansion / penetration experiments to measure intrinsic material properties, or dynamic tests to determine viscoelastic parameters (DTMA), to detect thermal events, and to separate overlapping transitions (MTMA™).

EXPANSION

Figure 1

Expansion measurements determine a material's coefficient of thermal expansion (CTE), glass transition temperature (Tg), and compression modulus. A flat-tipped standard expansion probe ***(Figure 1)*** is placed on the sample (a small static force may be applied), and the sample is subjected to a temperature program. Probe movement records sample expansion or contraction. This mode is used with most solid samples. The larger surface area of the macro-expansion probe ***(Figure 2)*** better facilitates analysis of soft or irregular samples, powders, and films

Figure 2

PENETRATION

Figure 3

Penetration measurements use an extended tip probe to focus the drive force on a small area of the sample surface ***(Figure 3)***. This provides precise measurement of Tg, softening, and melting behavior. It is valuable for characterizing coatings without their removal from a substrate. The probe operates like the expansion probe, but under a larger applied force. The hemispherical probe ***(Figure 4)*** is an alternate penetration probe for softening point measurements in solids.

Figure 4

TENSION

Figure 5

Figure 6

Tension studies of the stress/strain properties of films and fibers are performed using a Film/Fiber probe assembly ***(Figure 5)***. An alignment fixture ***(Figure 6)*** permits secure, and reproducible, sample positioning in the clamps. The clamped sample is placed in tension between the fixed and moveable sections of the probe assembly. Application of a fixed force is used to generate stress/strain and modulus information. Additional measurements include Tg, softening temperatures, cure, and cross-link density. Dynamic tests (e.g. DTMA, MTMA™) in tension can be performed to determine viscoelastic parameters *(e.g., E', E'', tan δ)*, and to separate overlapping transitions.

3-POINT BENDING

In this bending deformation (also known as flexure), the sample is supported at both ends on a two-point, quartz anvil atop the stage ***(Figure 7)***. A fixed static force is applied vertically to the sample at its center, via a wedge-shaped, quartz probe. Material properties are determined from the force and the measured probe deflection. This mode is considered to represent "pure" deformation, since clamping effects are eliminated. It is primarily used to determine bending properties of stiff materials (e.g., composites), and for distortion temperature measurements. Dynamic (DTMA) measurements are also available with the Q400EM, where a special low-friction metallic anvil replaces the quartz version.

Figure 7

SPECIALTY PROBE / FIXTURE KITS

Additional sample measurement probes and fixtures are available for use with both the Q400 and Q400EM in specialty TMA applications. These include:

Dilatometer Probe Kit **–** for use in volume expansion coefficient measurements

Parallel Plate Rheometer **–** for the measurement of low shear viscosity of materials (10 to 10^7 Pa.s range) under a fixed static force.

The expansion, macro-expansion, and penetration probes are supplied with the Q400. These probes, plus the flexure probe, and the low-friction bending fixture, are included with the Q400EM module. Data analysis programs relevant to each of the measurements described are provided in our Thermal Advantage™ for Q Series™ software.

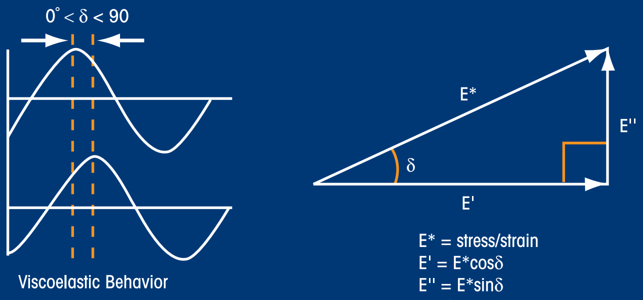
TMA THEORY

TMA measures material deformation under controlled conditions of force, atmosphere, time, and temperature. Force can be applied in compression, flexure, or tension modes using probes previously described. TMA measures intrinsic material properties *(e.g., expansion coefficient, glass transition temperature, Young's modulus)*, plus processing / product performance parameters *(e.g., softening points)*. These measurements have wide applicability, and can be performed by the Q400/Q400EM.

TMA can also measure polymer viscoelastic properties using transient *(e.g., creep, stress relaxation)* or dynamic tests. These require the Q400EM module. In creep, a known stress is applied to the sample, and its deformation is monitored. After a period, the stress is removed, and strain recovery is recorded. In stress relaxation, a fixed strain is applied, and stress decay is monitored.

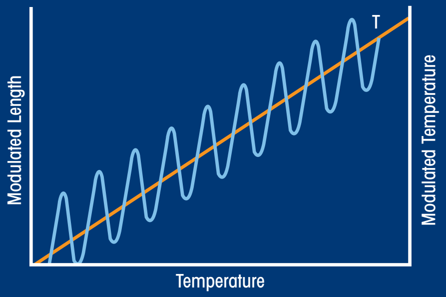
In Dynamic TMA (DTMA), a known sinusoidal stress and linear temperature ramp are applied to the sample, and the resulting sinusoidal strain, and sine wave phase difference (δ), are measured ***(Figure 8)***. From this data, storage modulus (E'), loss modulus (E''), and tan δ (E''/E') are calculated as functions of temperature, time, or stress.

FIGURE 8



$E^* = \text{stress/strain}$
$E' = E^* \cos\delta$
$E'' = E^* \sin\delta$
$\tan\delta = E''/E'$

FIGURE 9



In Modulated TMA™ (MTMA™), the sample experiences the combined effects of a linear ramp, and a sinusoidal temperature of fixed amplitude and period ***(Figure 9)***. The net signals, after Fourier transformation of the raw data, are total displacement and change in thermal expansion coefficient. Both can be resolved into their reversing and non-reversing component signals. The reversing signals contain events attributable to dimension changes, and are useful in detecting related events *(e.g., Tg)*. The non-reversing signals contain events that relate to time dependent kinetic processes *(e.g., stress relaxation)*.

Modes of Operation

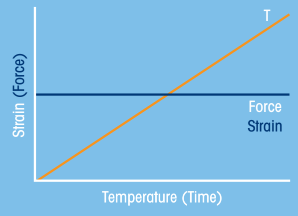
The Q400 and 400EM operating modes permit multiple material property measurements. The Q400 features the Standard mode, while the Q400EM additionally offers Stress/Strain, Creep, Stress Relaxation, Dynamic TMA, and Modulated™ TMA modes.

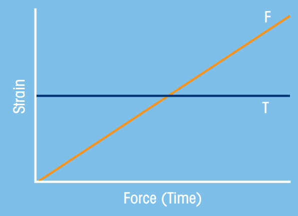
Standard Mode (Q400/Q400EM)

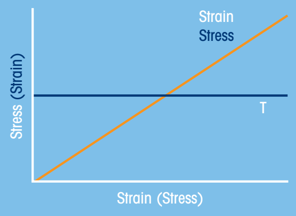
Temperature Ramp: Force is constant, and displacement is monitored under a linear temperature ramp. Provides intrinsic property measurements.

Isostrain: Strain is constant, and the force required to maintain it is monitored under a temperature ramp. Permits assessment of shrinkage forces in films/fibers.

Force Ramp: Force is ramped, and strain measured at constant temperature to generate force/displacement plots, and modulus information.





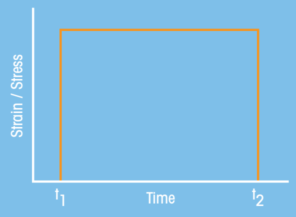


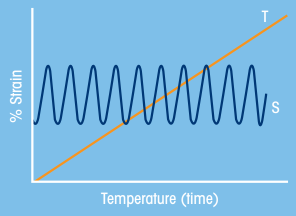
Stress/Strain Mode (Q400EM)

Stress or strain is ramped, and the resulting strain or stress is measured at constant temperature. Both provide stress / strain plots and related modulus information.

Creep/Stress Relaxation Modes (Q400EM)

In Creep, stress is held constant, and strain is monitored. In Stress Relaxation, strain is held constant, and stress decay is monitored. Both are transient tests used to assess material deformation and recovery properties.



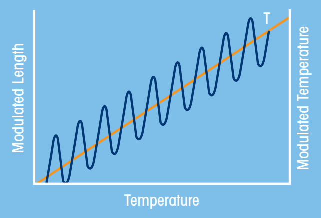


Dynamic TMA Mode (Q400EM):

A sinusoidal force (stress) is applied during a temperature ramp. Analysis of the resulting strain and phase data provides viscoelastic property parameters (e.g., E', E'' tan δ).

Modulated TMA Mode (Q400EM):

Temperature is programmed linearly, and simultaneously modulated at constant stress to generate signals relating to total displacement, CTE, and their reversing and non-reversing components. These permit detection of thermal transitions, and separation of overlapping events (e.g., Tg and stress relaxation).



APPLICATIONS

INTRINSIC AND PRODUCT PROPERTY MEASUREMENTS

Figure 10 shows expansion and penetration probe measurements of Tg, and softening point of a synthetic rubber using a temperature ramp at constant force. The large CTE changes in the expansion plot indicate the transition temperatures. In penetration, they may be detected by the sharp movement of the loaded probe into the changing material structure.

FIGURE 10

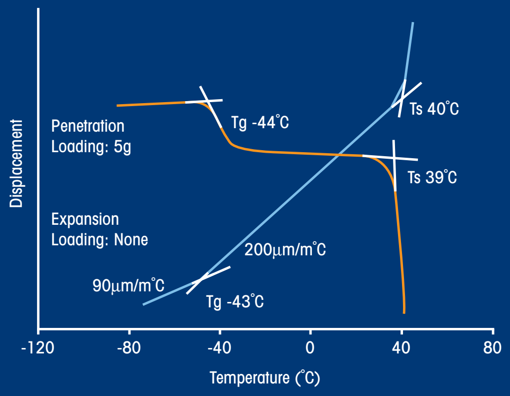
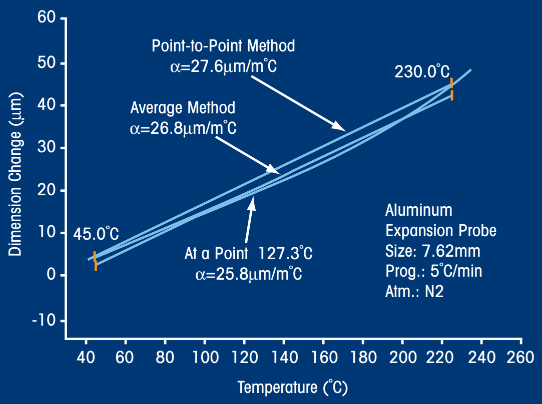


FIGURE 11



ACCURATE COEFFICIENT OF THERMAL EXPANSION (CTE) MEASUREMENTS

Figure 11 demonstrates the use of the expansion probe to accurately measure small CTE changes in an aluminum sample over a 200°C temperature range. Advantage™ software permits analysis of the curve slope using an “at point”, “straight line” or “best fit” method to compute the CTE (α) at a selected temperature, or over a range.

MATERIAL PERFORMANCE AND SELECTION

Figure 12 is an example of a 3-point bending mode (flexure probe) experiment on a polyvinyl chloride (PVC) sample, using the ASTM International Test Method E2092 to determine the distortion temperature. This test specifies the temperature at which a sample of defined dimensions produces a certain deflection under a given force. It has long been used for predicting material performance.

FIGURE 12

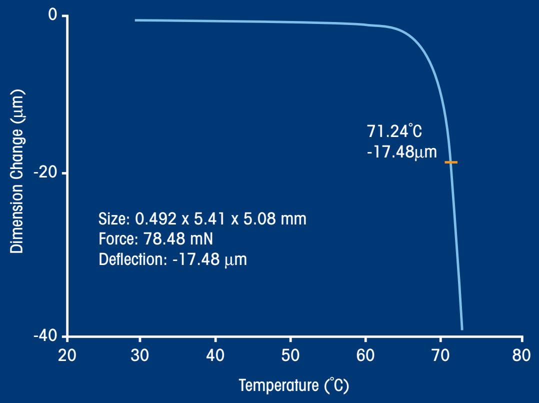
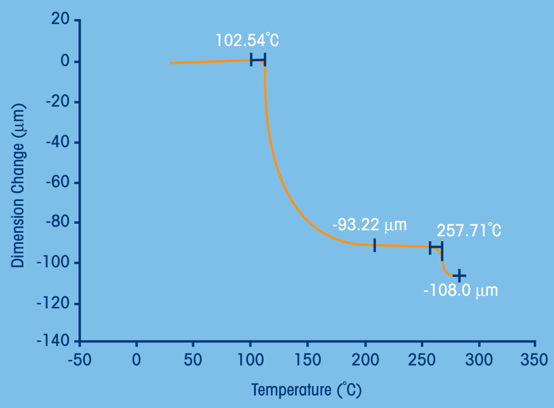


FIGURE 13



MULTILAYER FILM ANALYSIS

Figure 13 shows a compression mode analysis, using a penetration probe, of a double layer PE / PET film sample, supported on a metal substrate. The sample temperature was linearly ramped from ambient to 275 °C at 5 °C/min. The plot shows probe penetrations of the PE layer (93.22 µm) at 102 °C, and the PET layer (14.78 µm) at 257 °C respectively.

FILM PROPERTY TESTING

Figure 14 illustrates a classic isostrain experiment, in the tension mode, on a food wrapping film. The film was strained to 20% at room temperature for 5 minutes, cooled to -50 °C and held for 5 more minutes, then heated at 5 °C/min to 40 °C. The plot shows the force variation required to maintain a set strain in the film. The test simulates its use from the freezer to the microwave.

FIGURE 14

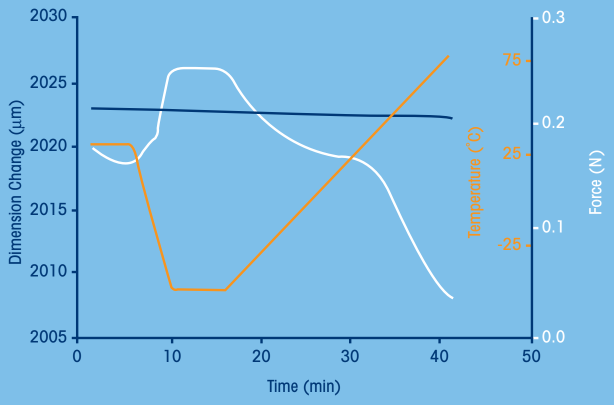
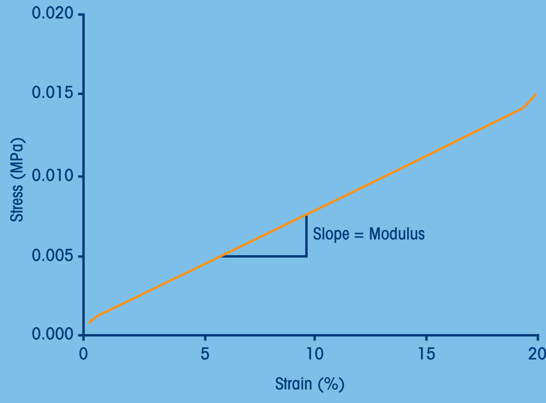


FIGURE 15



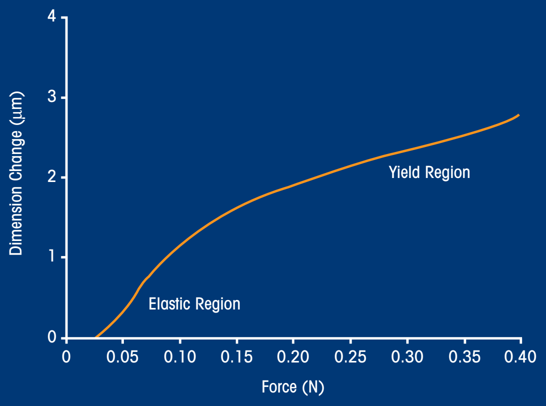
FILM TENSILE TESTING

Figure 15 displays a strain ramp experiment, at a constant temperature, on a proprietary film in tension. The plot shows an extensive region where stress and strain are linearly related, and over which a tensile modulus can be directly determined. The results show the ability of the Q400EM to function as a mini tensile tester for films and fibers.

FIBER STRESS/STRAIN MEASUREMENTS

Stress/strain measurements are widely used to assess, and compare, materials. ***Figure 16*** shows the different regions of stress/strain behavior in a polyamide fiber (25 μm) in tension, when subjected to a force ramp at a constant temperature. The fiber undergoes instantaneous deformation, retardation, linear stress/strain response, and yield elongation. Other parameters (e.g., yield stress; Young's modulus) can be determined.

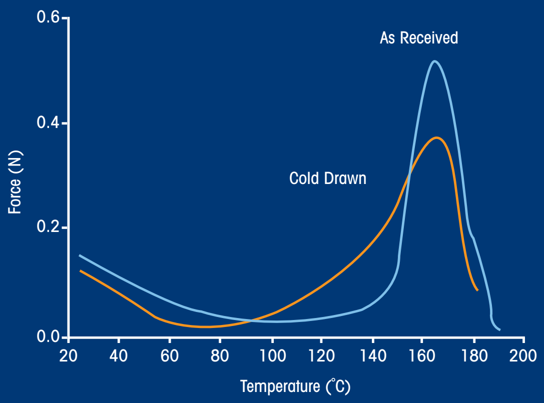
FIGURE 16



THERMAL STRESS ANALYSIS OF FIBERS

Figure 17 displays a tension mode experiment, using a temperature ramp at a constant strain (1%), to perform a stress analysis on a polyolefin fiber, as received, and after cold drawing. The plot shows the forces needed to maintain the set strain as a function of temperature. The data has been correlated with key fiber industry, processing parameters, such as shrink force, draw temperature, draw ratio, elongation at break, and knot strength.

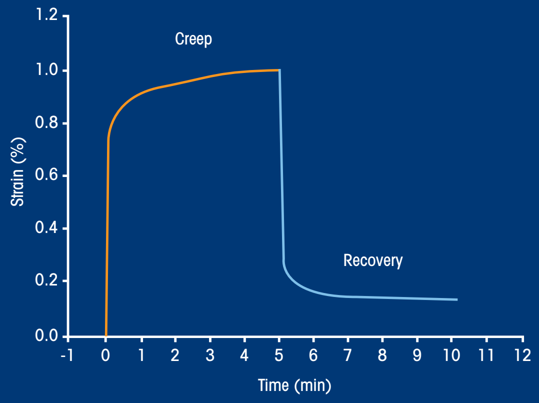
FIGURE 17



CREEP ANALYSIS

Creep tests help in materials selection for end-uses where stress changes are anticipated. ***Figure 18*** illustrates an ambient temperature creep study on a polyethylene film in tension. It reveals the instantaneous deformation, retardation, and linear regions of strain response to the set stress, plus its recovery with time on stress removal. The data can also be plotted as compliance, and recoverable compliance, versus time.

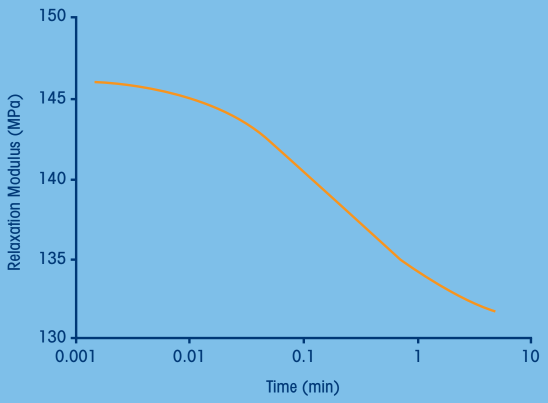
FIGURE 18



STRESS RELAXATION ANALYSIS

Figure 19 shows a stress relaxation test in tension on the same polyolefin film used for the creep study. A known strain is applied to the film, and maintained, while its change in stress is monitored. The plot shows a typical decay in the stress relaxation modulus. Such tests also help engineers design materials for end uses where changes in deformation can be expected.

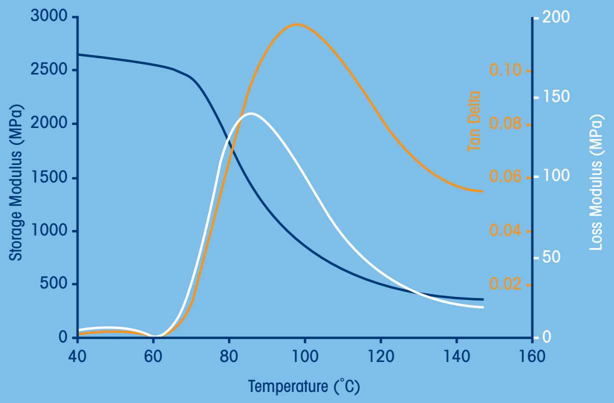
FIGURE 19



VISCOELASTIC PROPERTY DETERMINATION – DYNAMIC TMA

Figure 20 illustrates a dynamic test, in which a semi-crystalline polyethylene terephthate (PET) film in tension is subjected to a fixed sinusoidal stress during a linear temperature ramp. The resulting strain and phase data are used to calculate the material's viscoelastic properties (E', E'', and tan δ). The plotted data shows dramatic modulus changes as the film is heated through its glass transition temperature.

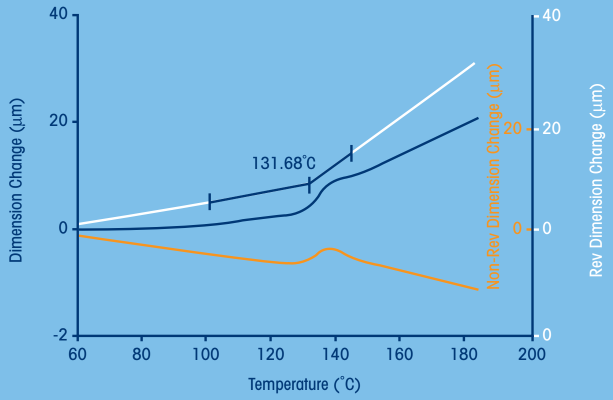
FIGURE 20



SEPARATING OVERLAPPING TRANSITIONS - MODULATED™ TMA

Figure 21 shows a MTMA™ study to determine the Tg of a printed circuit board (PCB). The signals plotted are the total dimension change, plus its reversing, and non-reversing components. The total signal is identical to that from standard TMA, but does not uniquely define the Tg. The component signals, however, clearly separate the actual Tg from the stress relaxation event induced by non-optimum processing of the PCB.

FIGURE 21

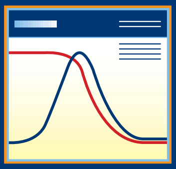


Thermal Advantage Software

A quality Thermal Analyzer requires flexible, intelligent software to empower it. No one believes this more than our software engineers, who have pioneered most of the features commonly seen in modern thermal analyzers. Q Series™ for Thermal Advantage™ software is Microsoft Windows® based, and expandable to meet growing user needs.

Thermal Advantage – Instrument Control

- ***Multitasking*** **–** conduct experiments and simultaneously analyzes data
- ***Multimodule*** **–** operates up to 8 modules simultaneously
- ***Wizards*** **–** guides and prompts in setting up experiments
- ***Real-Time Plot*** **–** provides a real-time display of the progress of the experiment
- ***Autoqueuing*** **–** permits pre-programmed set-up of planned experiments
- ***Autoanalysis*** **–** permits pre-programmed data analysis of planned experiments
- ***On Line Help*** **–** provides extensive, context sensitive, assistance
- ***Abort Feature*** **–** terminates a test upon attaining a specified value *(e.g., CTE)*



Universal Analysis 2000 – Data Analysis

- ***Single Software Package*** **–** analyzes data from all TA Instruments modules
- ***Picture-in-a-Picture*** **–** provides easy one plot analysis of large and small events
- ***Real-Time Data Analysis*** **–** analyzes data "as it arrives"
- ***Ability to create up to 20 simultaneous curve overlay plots***
- ***Custom Report Generation*** **–** within UA 2000 using Microsoft Word™ & Excel™ templates
- ***Saved Analysis*** **–** for quick retrieval of previously analyzed data files

WHY TA INSTRUMENTS

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

SALES AND SERVICE

We pride ourselves in the technical competence and professionalism of our sales force, whose only business is thermal analysis and rheology. TA Instruments is recognized worldwide for its prompt, courteous, and knowledgeable service staff. Their specialized knowledge and experience are major reasons why current customers increasingly endorse our company and products to their worldwide colleagues.

INNOVATIVE ENGINEERING

TA Instruments is the recognized leader for supplying innovative technology, investing twice the industry average in research and development. Our new Q Series™ Thermal Analysis modules are the industry standard. The Q400 TMA provides innovative technology suitable for research as well as QC laboratories. The Q400EM includes Dynamic TMA and also Modulated TMA™, a technique unavailable from other manufacturers.

QUALITY PRODUCTS

All thermal analyzers and rheometers are manufactured to ISO 9002 procedures in our New Castle, DE (USA) or our Leatherhead, UK facilities. Innovative flow manufacturing procedures and a motivated, highly skilled, work force ensure high quality products with industry leading delivery times.

TECHNICAL SUPPORT

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

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