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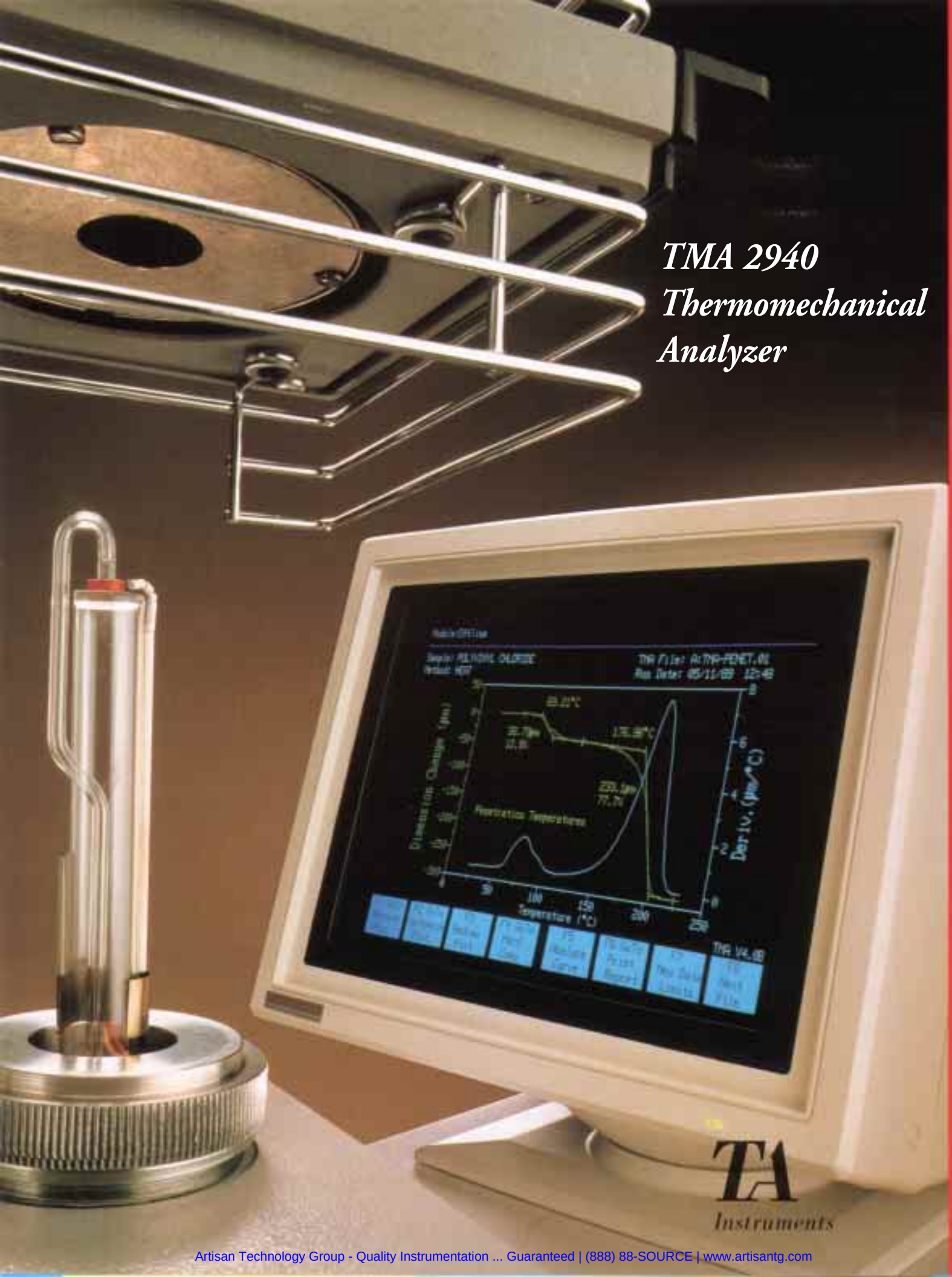
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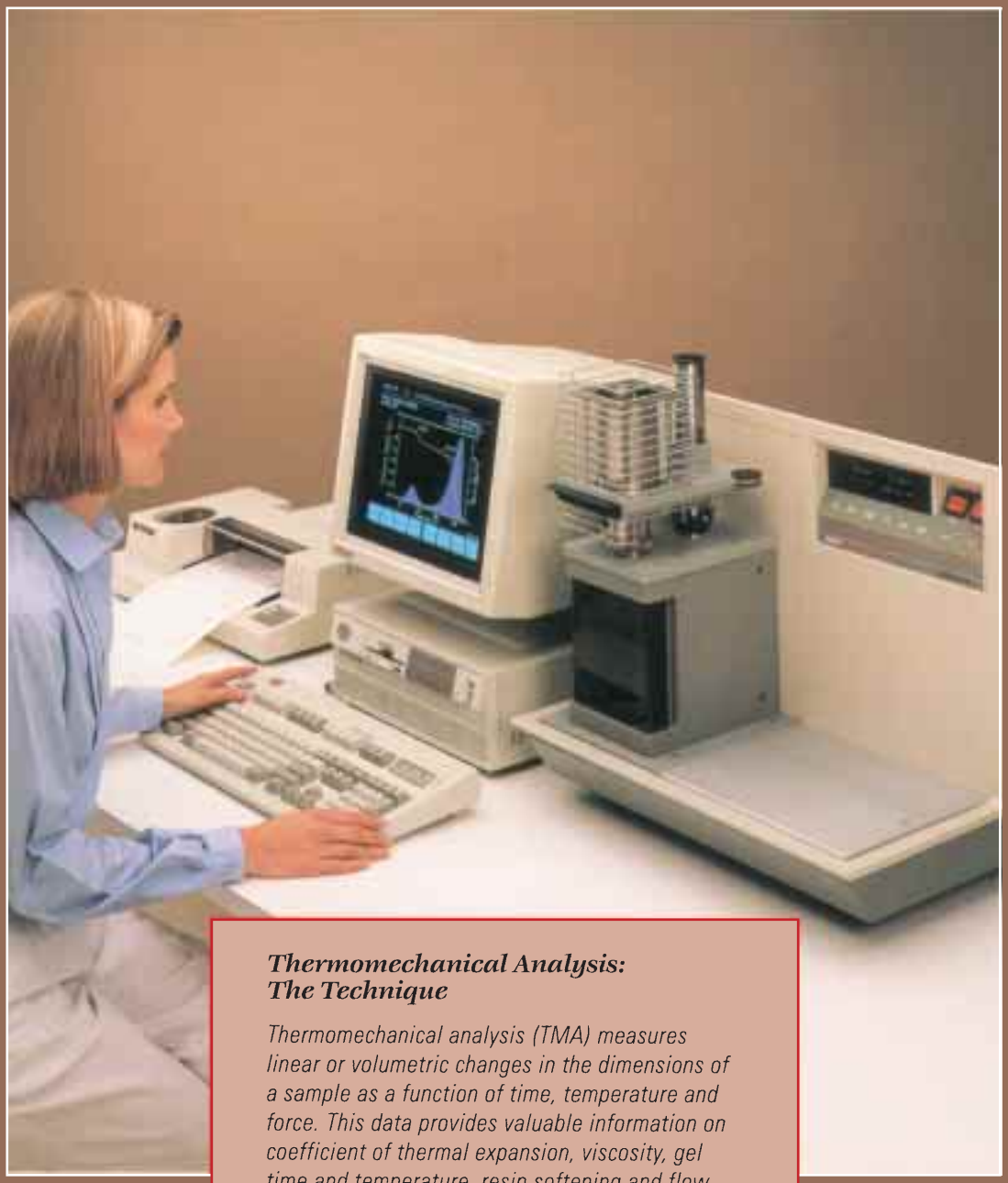
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Thermomechanical Analysis: The Technique

Thermomechanical analysis (TMA) measures linear or volumetric changes in the dimensions of a sample as a function of time, temperature and force. This data provides valuable information on coefficient of thermal expansion, viscosity, gel time and temperature, resin softening and flow, delamination temperature, glass transition temperature, modulus, and creep/stress relaxation. The technique is especially suited for studying the performance characteristics of polymeric materials, including thermoplastics, thermosets, elastomers, laminates, composites, films, fibers, adhesives and coatings. It is widely used for quality control, process optimization and trouble-shooting, as well as for research and development.

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TA Instruments TMA 2940 Thermomechanical Analyzer

The TA Instruments TMA 2940 brings new levels of performance, automation, flexibility and utility to the technique of thermomechanical analysis. It is accurate, fast and convenient to use, and generates highly reproducible, quantitative results. Major components of the instrument are shown schematically in Figure 2.

Improvements over previous generations of TA Instruments and competitive thermomechanical analyzers include mechanical and electronic automation features that will appeal particularly to those laboratories in which high throughput is important. Sample handling is easy and convenient, and minimizes the possibility of damaging the quartz stage and probe. The furnace assembly is keyed for precise alignment, and can be raised and lowered automatically or manually. In the raised position, it can be moved aside, providing ready access for stage/probe installation and sample loading. Movement of the installed probe relative to the sample is controlled electomechanically in response to push-button command of the operator. Sample measurement before a run is performed automatically after operator initiation. Auto zeroing is an option that can be programmed as one of the start-of-run conditions.

The electronics assure precise heat control with minimum drift, which helps give the instrument its high sensitivity. Five-point calibration capability helps optimize the accuracy of temperature measurements. The operating software automatically applies a quartz expansion factor to all sample dimension measurements.

Experiments involving furnace preheating are possible because furnace movement is mechanically driven and can be programmed as an experiment segment. When the ''preheat'' option is selected, the furnace is equilibrated in the ''up'' position, then automatically lowered over the sample, and an isothermal segment is initiated.

Seven interchangeable standard and special-purpose probes help give the TMA 2940 its great versatility. Operating modes include expansion, penetration, dilatometry, and parallel plate rheometry, plus tension modes for film and fiber stress/strain studies, fiber tension spectrometry and stress relaxation studies.

The operating software also offers end-of-run programmability. Options include automatic raising of the furnace and/or probe, and the introduction of forced air for rapid furnace cooling. This facilitates sample handling and clean-up, and minimizes turnaround time between experiments.

A keypad/display unit on the module offers operating convenience by allowing local control and monitoring of module functions. Push-button controls include experiment start/stop, furnace and probe positioning, probe zeroing, and sample length measurement. The display, controllable by a scroll key, provides real-time reports on a variety of experiment conditions.

What TMA Can Tell You

TMA experiments provide essential information that can help select the best materials for an application, predict product performance, and improve quality. The technique is particularly useful for determining:

Compatibility of materials that must function together: e.g., coatings and their substrates, adjacent layers of laminates, resins or elastomers and their reinforcements or fillers, seals or encapsulants and the mechanical systems they protect.

Suitability of materials for use in harsh environments and temperature extremes: e.g., brake linings, automotive gaskets, window seals, solder joints, adhesives, and protective coatings.

Physical characteristics and mechanical properties of materials, including films and fibers.

Optimum processing conditions for manufacturing efficiency, economy and product quality, including the ability to monitor rate and degree of cure of polymers.

Uses of TMA

Research and Development

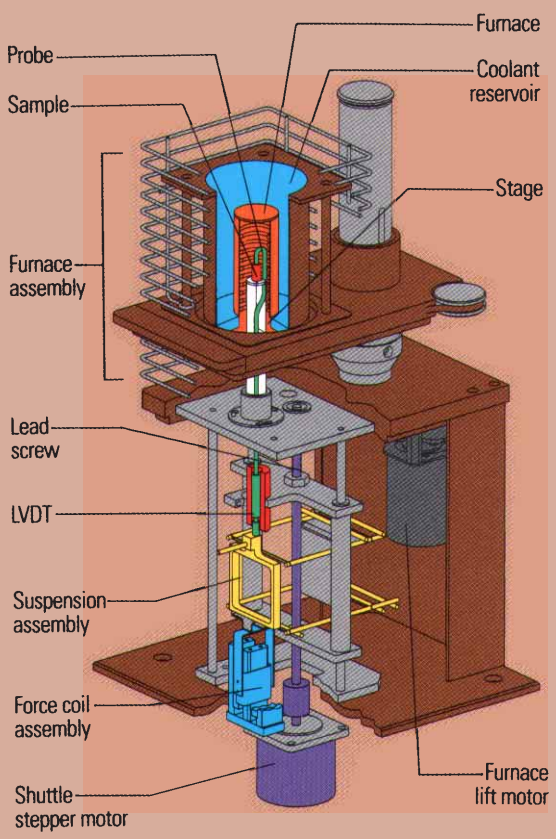
- Theoretical research on new materials and processes
- Materials selection
- Formulation optimization
- Applications development
- End-use performance prediction
- Competitive product evaluation

Quality Control/Assurance

- Vendor certification
- Incoming/outgoing material consistency
- Process optimization
- Finished product performance
- Troubleshooting

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Figure 2
TMA 2940's electromechanical system.



Principles of Operation

The value of thermomechanical analysis stems from its ability to measure linear or volumetric changes in samples as they are subjected to heat and mechanical distortion. The TMA 2940 components that create the environments for such phenomena and make the measurements are shown in Figure 2.

The heart of the TMA 2940 is a movable-core linear variable differential transformer (LVDT) whose output is proportional to the linear displacement of the core caused by changes in sample dimensions. Force is applied by an electromechanical coil, and the heat by a precisely controlled low-mass furnace. The sample chamber, located in the core of the furnace, also has provisions for cooling and atmosphere control. A thermocouple adjacent to the sample (Figure 3) assures accurate measurement of sample temperature. The final output is a plot of dimensional change and/or its derivative versus time, temperature, or force.

The versatile TMA 2940 is capable of operating in a variety of modes, using several probe/stage configurations, as illustrated on page 5. They are:

Compression Modes

Expansion probes are designed primarily to measure the coefficient of thermal expansion (CTE), glass transition temperature, and compression modulus of polymeric materials. The flat-tipped standard probe (Figure 3) is used for most solid materials. The macroexpansion probe (Figure 4), with its larger surface area, is more effective for soft or irregular samples, powders, frozen liquids, and films.

Penetration: The extension on the tip of the penetration probe (Figure 5) concentrates the force on a small area of the sample's surface for precise measurement of softening and melting points. It also can be used to examine coatings and films without removal from the substrate. An optional probe with a hemispherical tip (Figure 6) offers an alternate means for obtaining softening point data.

Dilatometry (Figure 7) is used to measure the volume coefficient of expansion of bulk or irregular-shaped materials. The sample, usually in granular or plug form, is contained in a vial surrounded by a silica filler and compressed by a macroexpansion probe. The 2940 software uses the output to plot its derivative which equals the linear CTE. With this data, a standard equation can be used to calculate the volumetric CTE.

Parallel Plate Rheometry (Figure 8) analyzes the low shear viscosity of materials over a range of 10 to 10^7 Pa. It is particularly useful for monitoring the cure of thermosets and composites, and for determining optimum processing times and temperatures. The sample is located between disks in an alignment cage. Force is applied by the macroexpansion probe.

Flexure: This device measures the deflection (bending) properties of stiff materials such as laminates and composites. The sample rests on a two-point anvil atop the stage, (Figure 9), while a wedge-shaped probe applies force. Properties are determined from force and deflection measurements.

Tension Modes

Films and Fibers: The stress/strain properties of films and fibers can be measured by using stainless steel clamps and a probe/stage assembly to accommodate them (Figure 10). Crimped-on aluminum spheres can be used with fiber samples (Figure 11), although clamps usually are preferred. An alignment fixture (Figure 12) helps attach the clamps to the sample.

The tension mode with an isostrain procedure is useful for investigating thermal history of fibers and determining the effects of processes such as draw ratio, heat setting and texturing. The isostrain capability also can be used to measure tension spectrometery and stress relaxation (decay).

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Features and Benefits

The TMA 2940 is designed with the operator in mind. It offers new levels of convenience, versatility, automation, and measurement sensitivity, while retaining all of the proven attributes of its predecessors. Key factors are:

Modular design, with seven interchangeable probes, two sample-support stages, and ancillary sample-testing assemblies.

Force control, with experiment programming for ramp, step, constant-load and isostrain conditions. This makes possible a variety of studies to measure the effects of force against time and temperature.

Isostrain capability. Through feedback control, the 2940 applies the force required to strain a sample to a specified percentage of initial size, and to maintain that strain throughout the experiment segment.

Force monitoring during an experiment.

Five-point temperature calibration for maximum accuracy. Operator can choose from one to five calibration standards.

Methods versatility, with ability to store up to 15 methods containing up to 60 segments selected from 18 available functions, including control of heating, cooling, force, environment and data handling.

Furnace preheating, making it possible to rapidly subject a sample to a high-temperature environment.

Automated calibration routines for force and instrument effects.

Local control at the module, including experiment start/stop, heater on/off, furnace and probe positioning, sample measurement, and real-time display of sample temperature and experiment status.

Sample-length measurement before each run is performed automatically after operator initiation.

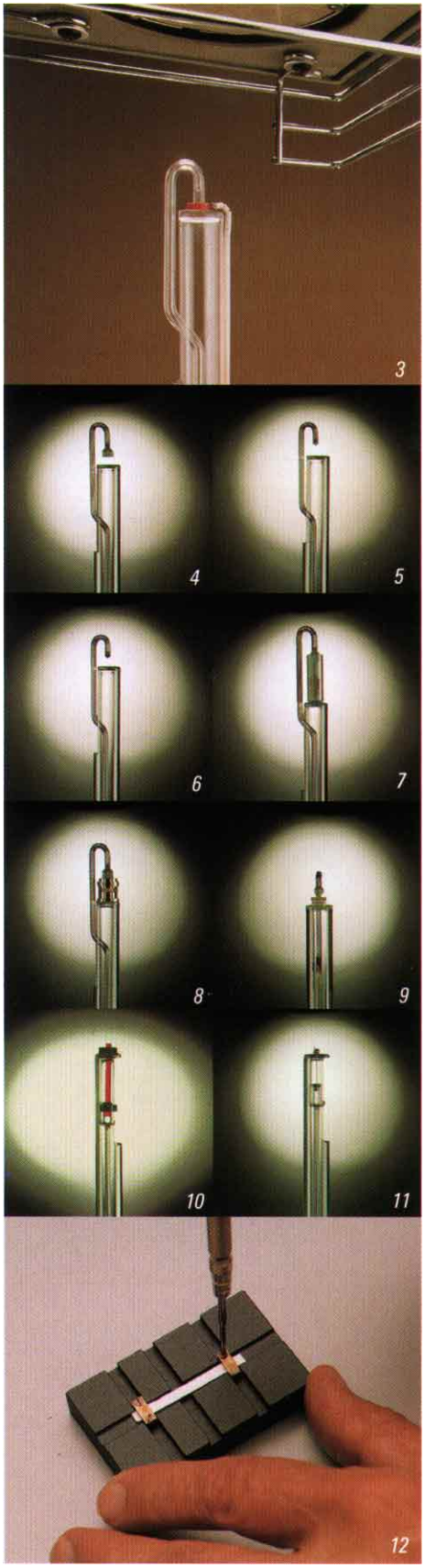
Auto zeroing as part of start-of-run programming.

Rapid turnaround, with programmable end-of-run conditions, including accelerated furnace cooling.

Unattended operation. Once an experiment has been programmed, all functional changes are made automatically. (Automatic changes in atmosphere require use of the optional Gas Switching Accessory.)

Analysis software: To obtain maximum value from TMA experiments, including quantification and interpretation of data, it is essential to use TA Instruments TMA data analysis software. Capabilities of the standard TMA data analysis program include:

- *Plotting versatility,* with a choice dimension change and its derivative, time, temperature or force on the "Y" axis, versus time, temperature or force on the "X" axis.
- *First derivative* generated simultaneously with base curve. This, together with the instrument's fast response and stable baseline, helps assure the high sensitivity needed to detect subtle transitions.
- *Coefficients of thermal expansion (CTE):* Positive or negative measurements at a single point, between two points, or fitted to a region.
- *Transition temperature measurements* that often are more sensitive than DSC, especially for filled materials and cured laminates.
- *Coating thickness* determination by analysis of penetration depth.
- *Process optimization* by evaluating data such as polymer blend effects, rate and degree of cure, processing time, and economics.



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Figure 13
Three-point calibrate-temperature curve

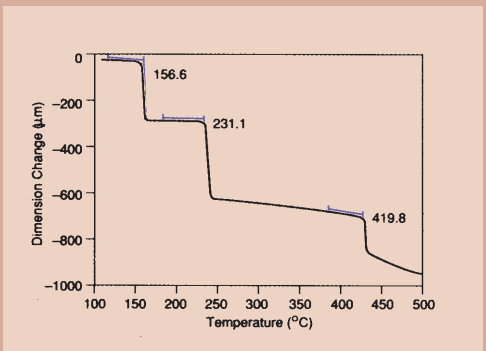


Figure 14
Measuring expansion of borosilicate glass

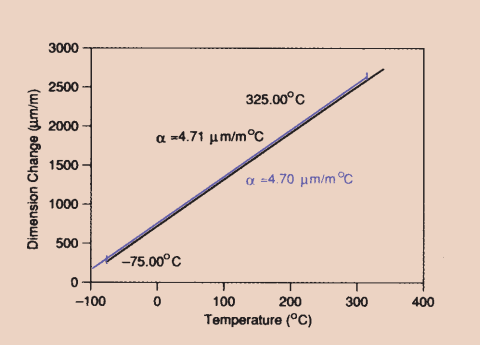


Figure 15
Measuring Tg of an elastomer by penetration

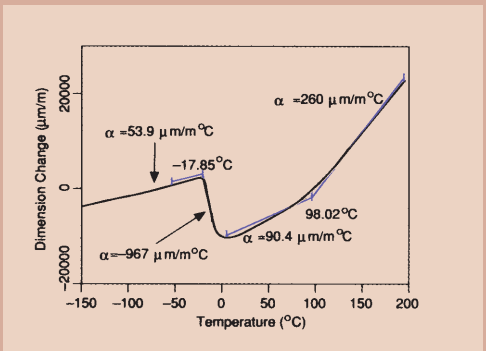
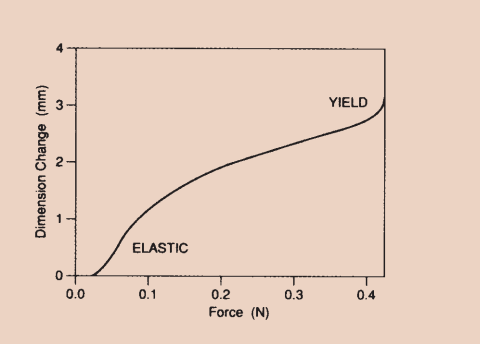


Figure 16
Measuring fiber stress/strain properties



Applications

The extended capabilities of the TA Instruments TMA 2940 as a valuable tool for materials characterization and quality control are illustrated by these applications. The examples were chosen to illustrate most of the TMA techniques, a sampling of the kinds of materials that can be examined, and many of the benefits inherent in the TMA 2940.

Multi-point Temperature Calibration

Calibration of the TMA's sample thermocouple is performed by comparing the observed melting points of standard materials with known values. The calibrated temperature curve for a "sandwich" of zinc, tin and indium is shown in Figure 13. The sample, contained in an aluminum pan to prevent stage contamination from the melted materials, was subject to ramp heating and constant force under an expansion probe. The curve shows three distinct transitions corresponding to the melting points. With this data, the 2940 software uses the cubic spline method to calculate corrections for any point on the measurement curve. Up to five standards can be used to optimize the calibration.

Two Methods of CTE Determination

Figure 14 illustrates the two principal methods for determining a material's coefficient of thermal expansion (CTE). In these experiments on borosilicate glass, dimension change in µm/m is plotted over a temperature range of −75°C to 325°C. Slope analysis of the resulting curve corresponds to the CTE. For materials exhibiting essentially linear expansion characteristics, as in this test, it is preferable to use the point-to-point method (a straight line connecting the chosen temperature limits). For materials whose expansion is non-linear, the second method (fitting a straight line to the data) must be used. This produces an average expansion value for the temperature range. In this example, both methods produced essentially the same results because of the linearity of the sample.

Glass Transition in Elastomers

Penetration of elastomers to yield accurate glass transition temperature readings is an excellent application for thermomechanical analysis. Figure 15 shows the results of an experiment subjecting an elastomer to a penetration load of 0.03 Newtons and a temperature range of −150°C to 200°C. The material shows a slight expansion below Tg, before allowing penetration at −17.85°C, resulting in a very marked glass transition. The expansion that takes place after Tg shows that the material is sturdy enough to resist further penetration, even in its rubbery state.

Fiber Stress/Strain Measurements

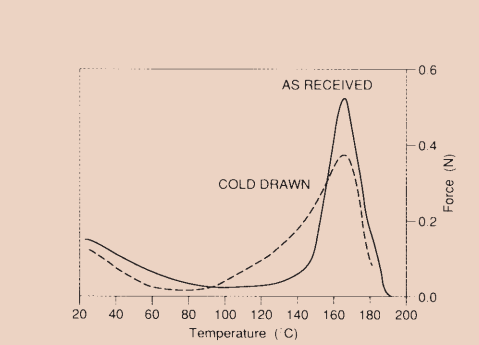
Stress/strain measurements are widely used to assess and compare materials. Although conventional physical testing devices can accommodate single-filament fibers, the results are difficult to obtain and accuracy is doubtful since the mass and inertia of the grips is much greater than the tensile strength of the fibers being evaluated. The clamping arrangement and the force range of the TMA 2940 system are more suitable for examining these fibers. With proper mounting in the fiber probe configuration to assure lack of end effects, TMA curves like that shown in Figure 16 for a 1 mil (25.4 µm) diameter polyamide filament can be obtained. From such a curve, it is possible to determine information about yield stress and Young's modulus in the elastic region.

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Thermal Stress Analysis

TMA should be an ideal technique for analyzing fibers since the measured parameters—dimension change, temperature, and stress—are major variables that affect fiber processing. Figure 17 shows the thermal stress analysis curves for a polyolefin fiber as received and after cold drawing. In this experiment, the fibers are subjected to initial strain (1% of initial length) and the force required to maintain that fiber length is monitored. Obviously, as the fiber tries to shrink, more force must be exerted to maintain constant length. The result is a direct measurement of the fiber's shrink force. Shrink force reflects the orientation frozen into the fiber during processing, which is primarily related to the amorphous portions of the fiber. Techniques which track fiber crystallinity, therefore, are not as sensitive a measure of processing conditions as TMA. In this case, the onset of the shrink-force peak indicates the draw temperature, while the magnitude of the peak is related to the fiber's draw ratio. It has been shown that the area under the shrink-force curve (from onset to maxima) can be correlated to properties such as elongation at break and knot strength. Other portions of the TMA thermal stress plot can yield additional information. For example, the initial decreasing slope is related to the fiber's expansion properties, and the appearance of secondary force peaks can be used to determine values such as heat set temperature in nylon.

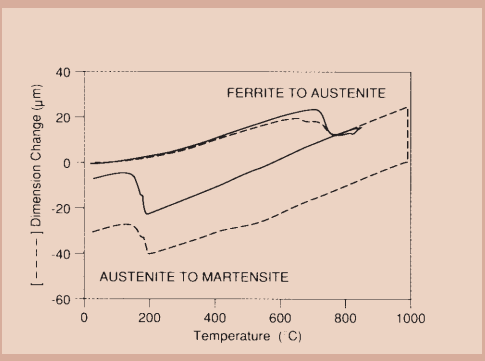
Figure 17
Thermal stress analysis of fibers



Crystalline Transitions in Metals

Stainless steel is an alloy used in many applications because it has the ability to recover after pseudoplastic deformation, a property which is dependent on the alloy's composition and its conditions of manufacture. Figure 18 shows TMA curves for two stainless steel samples of the same composition while being subjected to different processing (heat/cool) cycles. The major dimension changes correspond to changes in the alloy's internal crystalline structure. The goal in these alloys is to maximize martensite formation. The solid curve tracks dimension change during ballistic heating to 850°C, followed by immediate cooling at 20°C/minute. The dashed curve reflects dimension change during ballistic heating to 1000°C, an isothermal hold at 1000°C for 3 minutes, and cooling at 20°C/minute. The larger austenite-to-martensite dimension change in the former case suggests lower temperature processing results in more martensite in the final structure.

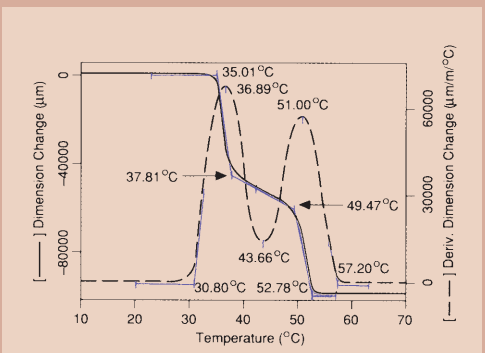
Figure 18
Determining crystalline transitions in metals



Characterizing Material Homogeneity by Penetration

The TMA 2940 in the penetration mode provides an easy, reliable means for characterizing a wax with a dual melting phase, as illustrated in Figure 19. The sample, contained in an aluminum pan, is subjected to a penetration force of 0.05 N and ramp heating at 5°C/min. Dimension change in µm/m and its derivative in µm/m°C are plotted against temperature. The step transitions in the melting curve show two distinct drops, at approximately 35°C and 49°C. The distinct peaks in the corresponding derivative curve, at approximately 37°C and 51°C, should give the operator confidence in the validity of the results.

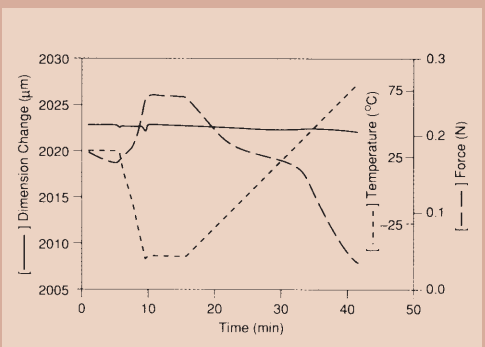
Figure 19
Evaluation of dual-melting wax



Using Isostrain to Test Film Properties

An experiment that explains why cling-type food wrapping film sometimes fails in the freezer is an excellent example of uses for the TMA 2940's isostrain capability. As shown in Figure 20, the sample was strained 20 percent at room temperature and held for 5 minutes, then cooled to −50°C and held for 5 minutes before being subjected to heating at 5°C/min. Interpretation of the data shows that maintaining a 20 percent strain requires significantly more force in the freezer: 0.25 Newtons at −50°C compared with only 0.15 Newtons at room temperature. Moral for the household user: overstretching cling-wrap does not improve its sealing power.

Figure 20
Why food wrap doesn't cling in the freezer



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Specifications

Dimensions: 66 cm (26″) H x 58.5 cm (23″) W x 45.5 cm (18″) D
Weight: 18 kg (60 lb)
Power Requirements: 115 VAC, 50/60 Hz, 10 amp
Temperature Range: —150°C to 1000°C
Sample Height (max): 25 mm (1″)
Sample Diameter (max): 10 mm (0.39″)
Sensitivity: 100 nanometers
Displacement Range: ±2.5 mm (0.10″)
Linearity: ±0.5%
Loading: 0.001 to 1.0 Newton (102 grams)
Atmosphere: Static or controlled flow with inert or reactive gases

TA Instruments Commitment

The TMA 2940 Thermomechanical Analyzer is designed and engineered to assure easy, reliable, trouble-free operation. It is supported by a full range of services, including an applications laboratory, publications, training courses, technical seminars, and a telephone Hotline for customer consultation. Highly qualified TA Instruments service personnel specialized in thermal analyzer maintenance and service are available throughout the world. All of these items reflect TA Instruments commitment to providing thermal analysis products and related support services that deliver maximum value for your investment.

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