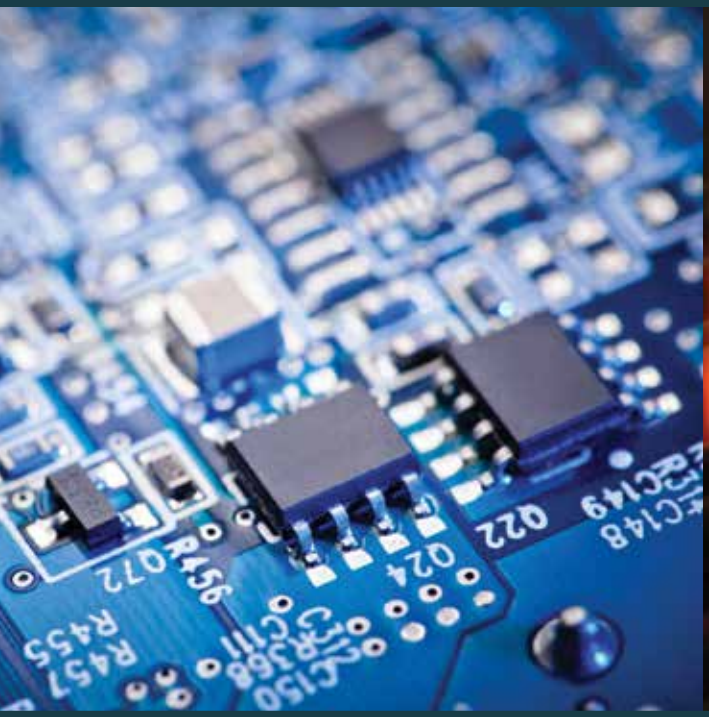
TA Instruments

DILATOMETRY

New Castle, DE USA
Lindon, UT USA
Hüllhorst, Germany
Shanghai, China
Beijing, China
Tokyo, Japan
Seoul, South Korea
Taipei, Taiwan
Bangalore, India
Sydney, Australia
Guangzhou, China
Hong Kong
Eschborn, Germany
Brussels, Belgium
Etten-Leur, Netherlands
Paris, France
Elstree, United Kingdom
Barcelona, Spain
Milano, Italy
Warsaw, Poland
Prague, Czech Republic
Sollentuna, Sweden
Helsinki, Finland
Copenhagen, Denmark
Chicago, IL USA
São Paulo, Brazil
Mexico City, Mexico
Montreal, Canada





dilatometry

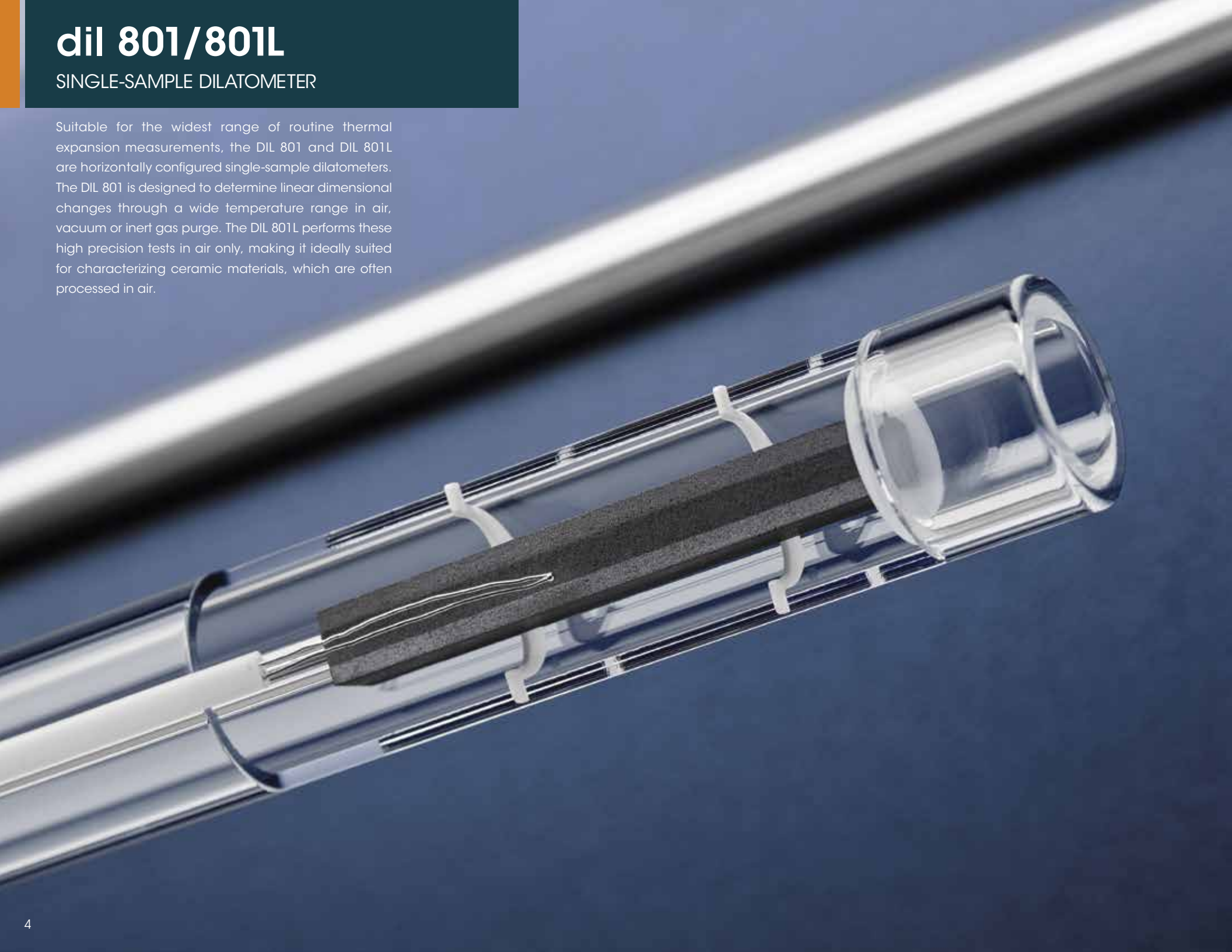
Every TA Instruments dilatometer precisely measures dimensional changes of a specimen brought about by changes in its thermal environment. Typical measurements include thermal expansion, annealing studies, determination of phase transitions and the glass transition, softening points, kinetics studies, construction of phase diagrams and sintering studies, including the determination of sintering temperature, sintering step and rate-controlled sintering. Investigation of processing parameters as reflected by dimensional changes of the material can be studied in great detail through exact duplication of thermal cycles and rates used in the actual process.

Each application of dilatometry has its own experimental requirements. That is why TA Instruments provides dilatometers in four basic types, each of which have flexibility of sample atmosphere, temperature and measurement control. Only TA Instruments can provide the right instrument to match your needs—no matter what your application may be.

dil 801/801L

SINGLE-SAMPLE DILATOMETER

Suitable for the widest range of routine thermal expansion measurements, the DIL 801 and DIL 801L are horizontally configured single-sample dilatometers. The DIL 801 is designed to determine linear dimensional changes through a wide temperature range in air, vacuum or inert gas purge. The DIL 801L performs these high precision tests in air only, making it ideally suited for characterizing ceramic materials, which are often processed in air.



	DIL 801	DIL 801L
Sample Length	0 to 50 mm	0 to 50 mm
Sample Diameter	max. 14 or 20 mm	max. 14 mm
Measurement System Holder	fused silica, Al_2O_3, sapphire graphite, or tungsten	fused silica, Al_2O_3
Change of Length	4 mm	4 mm
Length Resolution	10 nm	20 nm
Temperature Resolution	0.05 °C	0.1 °C
CTE Accuracy	0.03 x 10^{-6} K^{-1}	0.05 x 10^{-6} K^{-1}
Atmosphere	air, inert, reducing, vacuum	air
Operation Mode	horizontal	horizontal
Temperature Range	-160 °C to 2300 °C according to furnace type	-160 °C to 1650 °C according to furnace type
Contact Force	0.02 N to 1.00 N, adjustable	0.02 N to 1.00 N, adjustable

dil 802/802L

DIFFERENTIAL DILATOMETER

For the highest precision and accuracy, the DIL 802/802L provides a true differential measurement in a horizontal dilatometer. Detecting only the difference between the sample and an inert reference specimen, the design of the DIL 802/802L negates the influence of system expansion on the sample measurement. This design is of particular benefit for dynamic temperature programs, such as those employed in rate-controlled sintering (RCS), and for experiments conducted at lower temperatures. The DIL 802/802L can also be converted for use as a single-sample dilatometer.

	DIL 802	DIL 802L
Sample Length	0 to 50 mm	0 to 50 mm
Sample Diameter	max. 7 or 10 mm after conversion to DIL 801: 14 or 20 mm	max. 7 mm after conversion to DIL 801L: 14 mm
Material of Sample Holder	fused silica, Al_2O_3, sapphire graphite, or tungsten	fused silica, Al_2O_3
Change of Length	4 mm	4 mm
Length Resolution	10 nm	20 nm
Temperature Resolution	0.05 °C	0.1 °C
CTE Accuracy	0.01 x 10^{-6} K^{-1}	0.03 x 10^{-6} K^{-1}
Atmosphere	air, inert, reducing, vacuum	air
Operation Mode	horizontal	horizontal
Temperature Range	-160 °C to 2300 °C according to furnace type	-160 °C to 1650 °C according to furnace type
Contact Force	0.02 N to 1.00 N, adjustable	0.02 N to 1.00 N, adjustable

dil 803/803L

DUAL-SAMPLE DILATOMETER

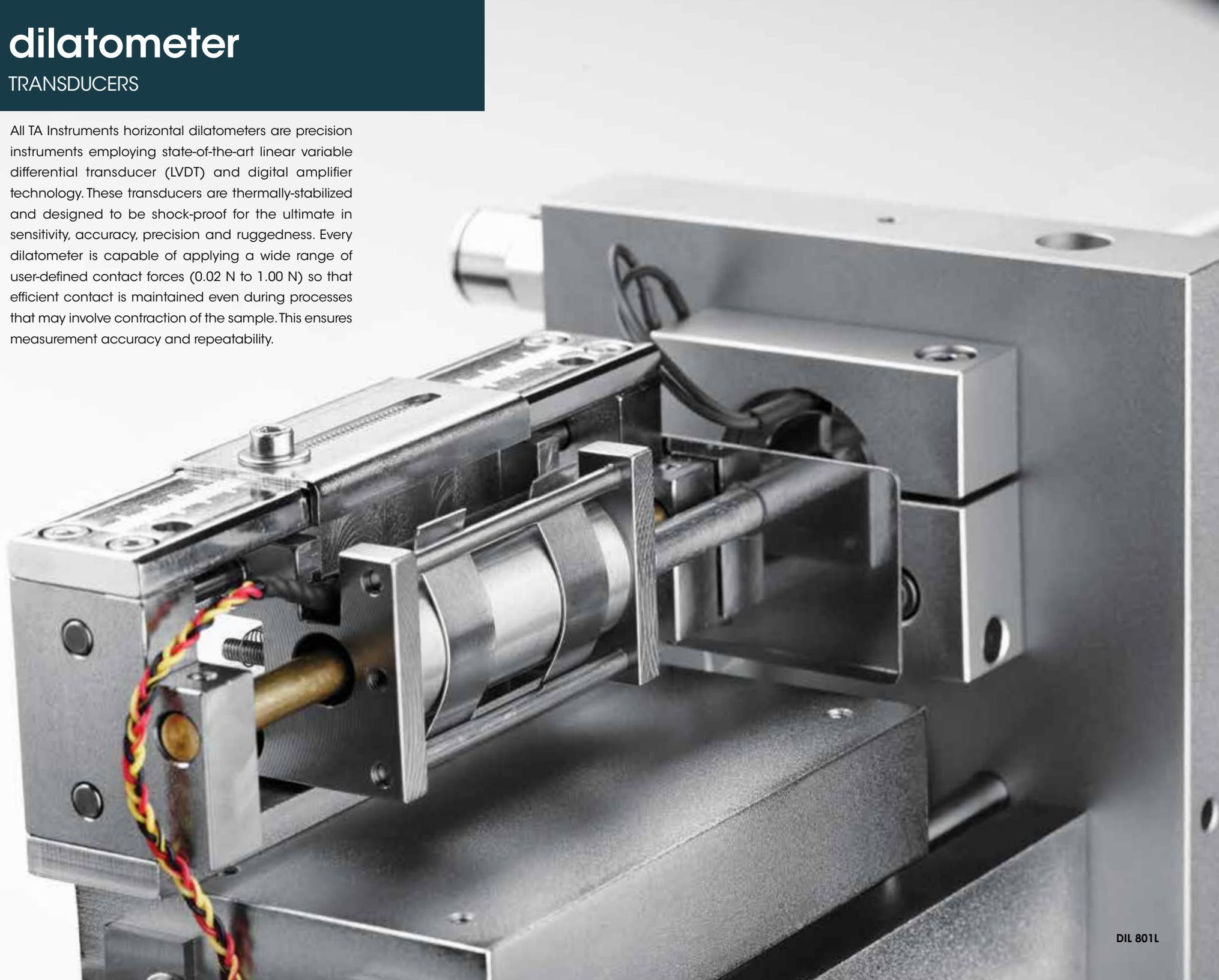
To maximize sample throughput, the DIL 803/803L offers dual-sample simultaneous operation in a horizontal dilatometer. The DIL 803/803L can also be operated as a differential system, using an inert reference specimen to reduce the influence of system expansion, increasing accuracy under dynamic temperature conditions. Combined with TA's easily interchanged furnaces that reduce cooling time between experiments, the DIL 803/803L maximizes productivity in any lab.

	DIL 803	DIL 803L
Sample Length	0 to 50 mm	0 to 50 mm
Sample Diameter	max. 7 or 10 mm after conversion to DIL 801: 14 or 20 mm	max. 7 mm after conversion to DIL 801L: 14 mm
Material of Sample Holder	fused silica, Al_2O_3, sapphire	fused silica, Al_2O_3
Change of Length	4 mm	4 mm
Length Resolution	10 nm	20 nm
Temperature Resolution	0.05 ˚C	0.1 ˚C
CTE Accuracy	0.03 x 10^{-6} K^{-1}	0.05 x 10^{-6} K^{-1}
Atmosphere	air, inert, reducing, vacuum	air
Operation Mode	horizontal	horizontal
Temperature Range	-160 ˚C to 1650 ˚C according to furnace type	-160 ˚C to 1650 ˚C according to furnace type
Contact Force	0.02 N to 1.00 N, adjustable	0.02 N to 1.00 N, adjustable

dilatometer

TRANSDUCERS

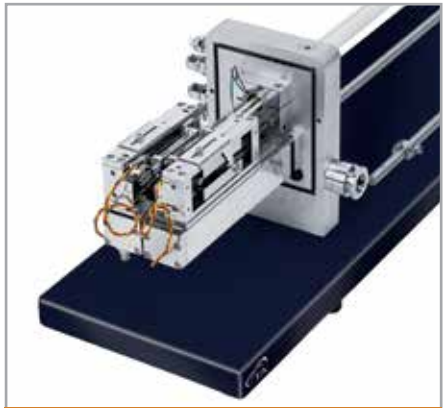
All TA Instruments horizontal dilatometers are precision instruments employing state-of-the-art linear variable differential transducer (LVDT) and digital amplifier technology. These transducers are thermally-stabilized and designed to be shock-proof for the ultimate in sensitivity, accuracy, precision and ruggedness. Every dilatometer is capable of applying a wide range of user-defined contact forces (0.02 N to 1.00 N) so that efficient contact is maintained even during processes that may involve contraction of the sample. This ensures measurement accuracy and repeatability.



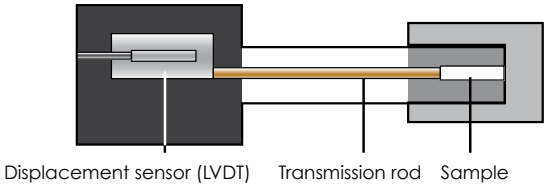
DIL 801L

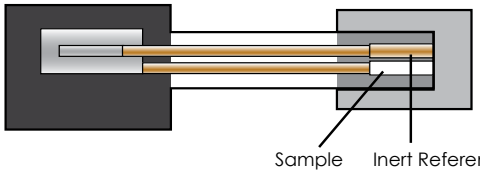
DIL 801 (Standard)

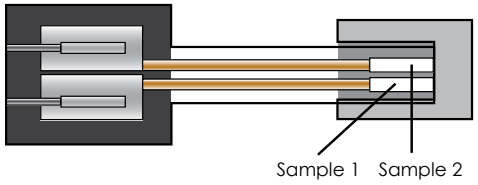
DIL 802 (Differential)



DIL 803 (Dual-Sample)



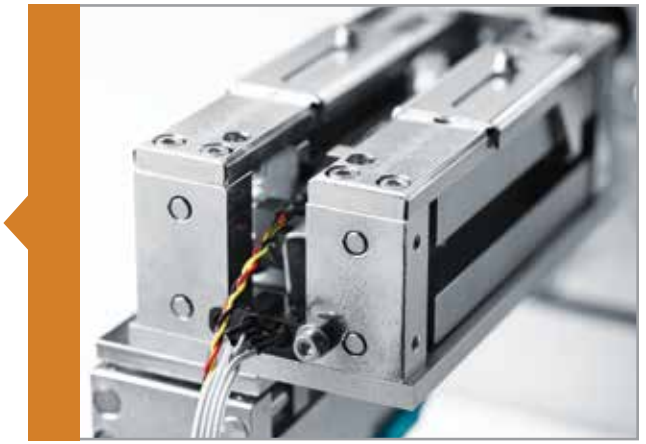




True Differential Measurement DIL 802

The DIL 802 features a true differential measurement design that maximizes precision and accuracy. Many two-sample dilatometers can operate in differential mode, in which the signals from two separate transducers are subtracted from one another. Unlike these "software differential" instruments, the DIL 802 is designed specifically for the high performance of true differential operation. At the heart of the DIL 802 is a single displacement transducer with an innovative measurement design that reduces noise and maximizes accuracy. The core of the differential transducer is coupled to the reference specimen while the coil of the transducer is coupled to the sample. The transducer's frame of reference moves with system expansion, leaving only the excess sample expansion to be measured. This results in:

- **Increased accuracy**
- **Reduced reliance on system calibration**
- **Increased temperature program flexibility**



dilatometer

FURNACE OPTIONS

Every TA Instruments horizontal dilatometer can be configured with a wide range of furnace options, depending on the temperature range requirements. Furnaces are easily interchangeable, providing the utmost in configuration flexibility. Multiple furnaces of the same type can also be used to increase sample throughput on a single instrument.

Temperature	-160 °C to 700 °C	20 °C to 1350 °C	20 °C to 1500 °C	100 °C to 1650 °C	20 °C to 2000 °C 300 °C to 2300 °C
Heating element	NiCr with sheath	CrAlFe	SiC	Noble metal	Graphite
Cooling medium	liquid nitrogen				
Thermocouple type	K	S	S	B	C or B
Pyrometer					Spectral or Two-color
Max. heating rate (K/min)	50	50	50	25	150
Max. cooling rate (K/min)	25	10	15	5	100
Temperature profile over 50 mm	±2 ˚C	±3 ˚C	±5 ˚C	±5 ˚C	±5 ˚C over 20 mm
Furnace cooling	air	air	water	air	water

-160 ˚C to 700 ˚C

20 ˚C to 1350 ˚C

20 ˚C to 1500 ˚C

100 ˚C to 1650 ˚C

20 ˚C to 2300 ˚C

Choosing a Dilatometer for Your Application

The success and accuracy of the dilatometric measurement depends greatly on proper instrument selection. There are substantial differences between the various configurations, each being better suited for a particular measurement and application.

Horizontal

- Simple, robust design – easy to use
- Best temperature uniformity
- Greatest flexibility

Vertical

- Best mode for samples which may shrink on heating, such as powdered metals or ceramics in sintering
- Vertical design ensures consistent push-rod contact
- Capable of additional TMA-type experiments

Non-contact Optical

- Excellent heating profile (above and below the sample)
- Best choice for irregularly shaped and soft samples
- Non-contact measurement eliminates push-rods and associated effects
- Absolute expansion measurement, competely independent of system

Softening Point Detection

Because material behavior is often not known when designing an experiment, the instrument control software includes automated softening point detection. Several conditions can be set to determine softening point and subsequent instrument behavior. This allows for reliable, unattended operation on unknown materials without risk of instrument damage.

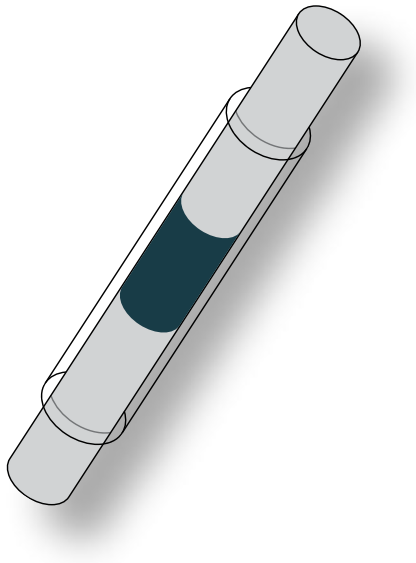
Testing Standards

TA Instruments dilatometers conform to all major standard test methods for dilatometry. These methods include:

ASTM C372	**ASTM E228**	**DIN 52328**
ASTM C531	**ASTM E831**	**DIN 53752**
ASTM C824	**DIN 51045**	**SEP 1680**
ASTM D696	**DIN 51909**	**SEP 1681**

Liquid and Paste Samples

The special liquid and paste cell allows for measurements to be made on high viscosity liquids, pastes and powders. Measurement accuracy is enhanced through a software-driven correction for dead volume and containment effects.



dil 811

VERTICAL DILATOMETER

The DIL 811 operates in a vertical orientation, making it ideal for the analysis of sintering, RCS (rate-controlled sintering), viscoelastic behavior and the determination of softening temperatures. The linear motor generates a constant force throughout the experiment, ensuring that contact is maintained with the sample regardless of dimensional change. The motor can also apply a dynamic force and viscoelastic properties may be determined. The high resolution inductive sensor (LVDT) ensures the best sensitivity to small dimensional changes.

	DIL 811
Sample Length	0 to 25 mm
Sample Diameter	max. 14 or 20 mm
Sample Holder Material	fused silica, Al_2O_3, graphite
Contact Force	0.01 N to 2.5 N
Frequency	0.05 Hz to 50 Hz
Change of Length	5 mm
Resolution ΔL, ˚C	10 nm, 0.05 ˚C
CTE Accuracy	0.05 x 10^{-6} K^{-1}
Atmosphere	inert gas, vacuum, air
Temperature Range	RT to 1500 ˚C 100 °C to 1650 ˚C RT to 2000 ˚C

modes of deformation

DIL 811

The DIL 811 offers all the deformation modes necessary to characterize a wide range of materials for thermal expansion and contraction, key transition temperatures, and mechanical properties. The available probe profiles include flat, hemispherical, pointed, and beam bending.

Flat

Expansion measurements determine a material's coefficient of thermal expansion (CTE), glass transition temperature (T_g), and compression modulus. A flat-tipped expansion probe is the most common mode of deformation and provides an accurate measurement of macroscopic dimensional change. This wide contact area is especially important for materials that may contract significantly, such as ceramics during sintering. The large contact area ensures that positive contact is maintained throughout the experiment and allows low stresses to be applied accurately and uniformly.

Pointed Penetration

Penetration measurements use an extended tip probe to focus the drive force on a small area of the sample surface. This provides precise measurement of glass transition (T_g), softening or melting behavior. It is valuable for characterizing coatings without their removal from a substrate or for measuring local behavior in a heterogeneous material. The probe operates like the Flat probe, but under a larger applied stress.

Hemispherical

Similar to the Pointed Penetration probe, the Hemispherical probe applies force on a small area. This provides an alternative to the Pointed Penetration probe with a nominally point-contact that is highly reproducible. Both the Pointed Penetration and Hemispherical probes are well-suited for measuring the compression modulus of medium to high stiffness materials, for which high applied stresses are desired.

3-Point Bending

In bending deformation (also known as flexure), the sample is supported at both ends on a two-point anvil atop the stage. A fixed static force is applied vertically to the sample at its center, via a wedge-shaped probe. This mode is considered to represent "pure" deformation, since clamping effects are eliminated. It is primarily used to determine bending properties of stiff materials, and for distortion temperature measurements. Dynamic measurements are also available with the application of sinusoidal or square wave force profiles.

Flat

Pointed Penetration

Hemispherical

3-Point Bending

modes of operation

The DIL 811 provides a full range of test modes for the most accurate dilatometry experiments. In addition, the DIL 811 has enhanced functions, more commonly associated with a thermomechanical analyzer (TMA), that can be performed with the utmost accuracy and over the widest range of temperatures.

Standard

In the most common mode of operation, a defined force is applied to the sample to establish and maintain contact while the sample is subjected to a predefined temperature program. This temperature program may include several segments of heating, cooling and isothermal conditions. This is the primary method by which coefficient of thermal expansion (CTE) is measured. Phase transitions are easily detected as the point at which the CTE changes, or when other large dimensional changes occur (such as in a softening point). Time and temperature-dependent kinetic events, such as sintering, can also be measured using the standard mode of operation. These kinetic processes can be distinguished from simple phase transitions by performing experiments at varying heating rates or operating in sequential isothermal steps. The outstanding temperature control and uniformity of the DIL 811 permits fine control of these temperature profiles.

Constant Force Control

While force is often applied statically, it may be programmed to change in a step-wise, or linear manner, throughout the course of an experiment. Fine control of the force over the wide range of 0.01 N to 2.5 N allows for adjustment of sintering pressure, as well as the direct measurement of mechanical properties (such as the modulus).

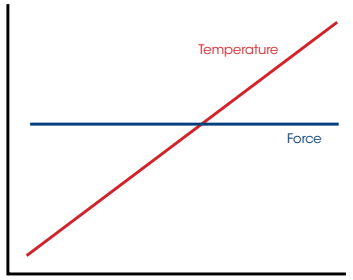
Dynamic Force Control

The DIL 811 can also apply dynamic loads to a sample during any temperature program. These dynamic loads can be applied in sinusoidal or square wave profiles and over a wide frequency range from 0.05 Hz to 50 Hz. Measurement under these conditions allows for the calculation of viscoelastic properties such as the storage modulus, loss modulus, and tan δ.

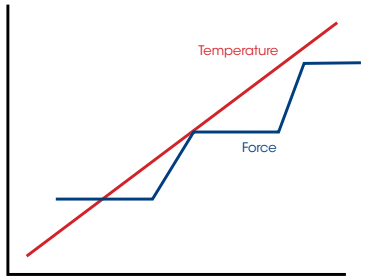
Rate-Controlled Sintering

In addition to preprogrammed temperature profiles, the DIL 811 can be operated under conditions of Rate-Controlled Sintering (RCS). Rate-Controlled Sintering software permits the user to define a target sintering (contraction) rate. The temperature profile is then adjusted to achieve this rate by increasing or decreasing the heating rate in real time.

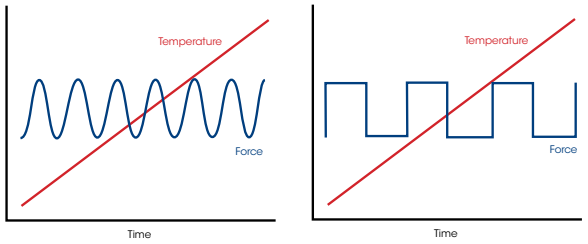
Standard



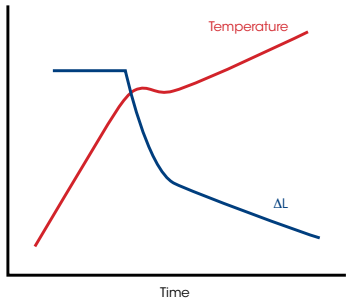
Constant Force Control



Dynamic Force Control



Rate-Controlled Sintering



dil 806

OPTICAL DILATOMETER

The DIL 806 optical dilatometer is an innovative[1] and versatile instrument for thermal expansion and contraction measurements. Sample length is measured entirely without contact, making the instrument ideally suited for thin, irregularly shaped and soft samples.

[1] US Patent # 7,524,105

	DIL 806
Sample Length	0.3 mm to 30 mm
Sample Height	max. 10 mm
Change of Length	max. 29 mm
Length Resolution	50 nm
Temperature Resolution	0.1 °C
CTE Accuracy	0.05 x 10^{-6} K^{-1}
Temperature Range	-150 °C to 600 °C RT to 900 °C RT to 1400 °C
Atmosphere	vacuum, inert gas, air DIL 806L: air only

technology

DIL 806

The DIL 806 Optical Dilatometer uses an innovative new measurement principle to make unconventional dilatometry experiments possible, and to improve many conventional tests.

Measurement Principle

The DIL 806 operates by the shadowed light method. In this method, the absolute size of a sample is measured in one direction by measuring the shadow cast by that sample on a high precision Charge-Coupled Device (CCD) detector. A high intensity GaN LED emits a plane of light, which is passed through a diffusion unit, and collimating lens to produce a highly uniform, short wavelength, plane of light. The sample blocks transmission of a portion of this light. This now-shadowed light is refined through a telecentric optical system and recorded by a high-resolution CCD. Digital edge detection automatically determines the width of the shadow, and therefore the dimension of the sample.

Absolute Measurement Advantages

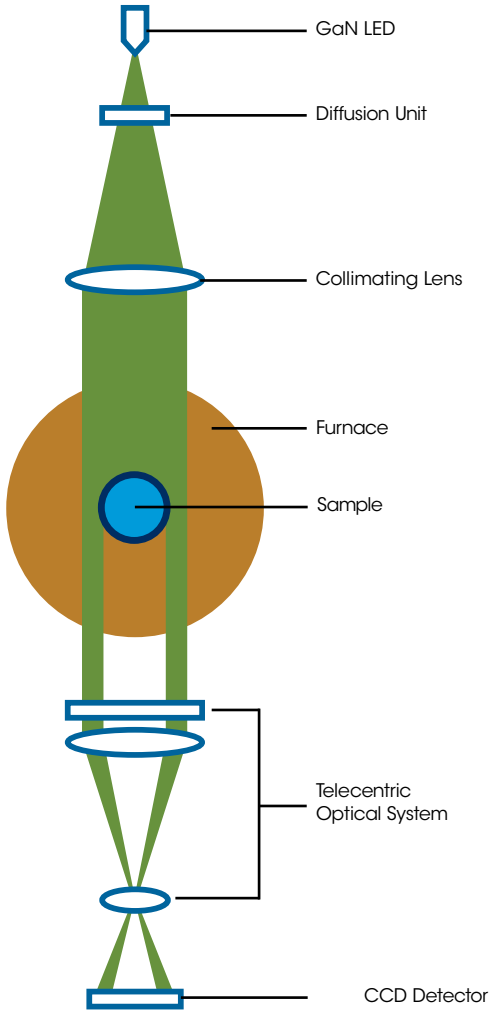
The measurement of the DIL 806 is an intrinsically absolute measurement, unaffected by system thermal expansion that accompanies the programmed changes in temperature. Only the sample is subjected to temperature excursions; both the light source and detector are well-isolated from these changes. Consequently, the measurement is absolute, not requiring the test-specific calibrations that are common with push-rod dilatometers.

Furnace Technology

The DIL 806 features an innovative plate-shaped furnace, which provides superior temperature uniformity and response time. The sample is positioned centrally within the wide planar heating element, which is much larger than the sample, preventing thermal gradients in the lateral direction. A similar heating element in the furnace lid is positioned immediately above the sample, minimizing vertical temperature gradients.

The furnace is capable of rapid heating speeds up to 100 °C/min and cooling times from 1400 °C to 50 °C in under 10 minutes. These rapid heating and cooling speeds enable high sample throughput or processes characterized by rapid changes of temperature.

The dynamic furnace response also makes the DIL 806 especially well-suited to Rate-Controlled Sintering experiments, which are supported by the instrument control software. This custom software package permits the user to define a target sintering (contraction) rate. The temperature profile is then adjusted to achieve this rate by increasing or decreasing the heating rate in real-time response to the sample behavior.



Non-Contact Measurement Benefits

The non-contact measuring system provides several advantages. Because no load is applied to the sample, even the softest materials can be tested with the highest precision. These samples may include thin films, or those materials that are inherently soft or experience a softening transition during the course of the experiment.

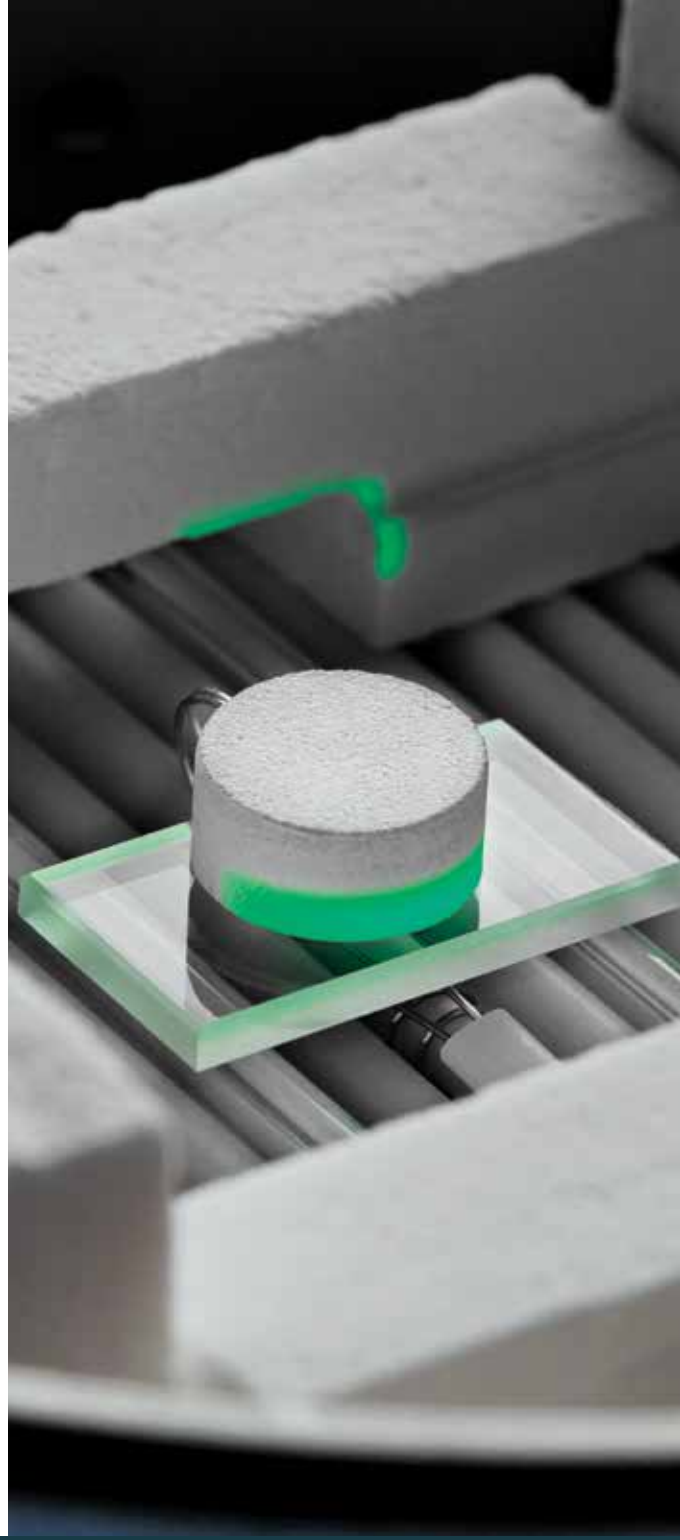
The absence of physical contact with the measuring system further enhances temperature uniformity. Push-rods can act as heat sinks, generating hot or cold spots at the point of contact with the sample. The DIL 806 is free of these contact points, ensuring that the entirety of the sample is at a uniform temperature throughout the experiment, regardless of experimental temperature profile.

Sample positioning is greatly simplified by the wide measurement area. Using a measurement area 30 mm wide, the instrument works equally well with a sample positioned anywhere in this range. This simplifies sample loading by removing strict restrictions on sample position.

Sample Types

The DIL 806 provides the maximum flexibility in sample type and preparation. The lack of push-rod contact removes the requirement for smooth or parallel sample faces. Irregularly shaped samples can be measured without difficulty. Thin films can easily be measured in their length or width direction and the DIL 806 operates equally well with optically opaque, translucent or transparent materials. A single sample can be measured in several directions, allowing for the facile identification of anisotropic thermal expansion in composites or other oriented materials.

Because the DIL 806 can accommodate many sample types and shapes, it is a natural complement to other measurements. The same specimen may be measured in the DIL 806 as is measured by dynamic mechanical analysis, the flash technique for thermal conductivity, surface hardness density and more.

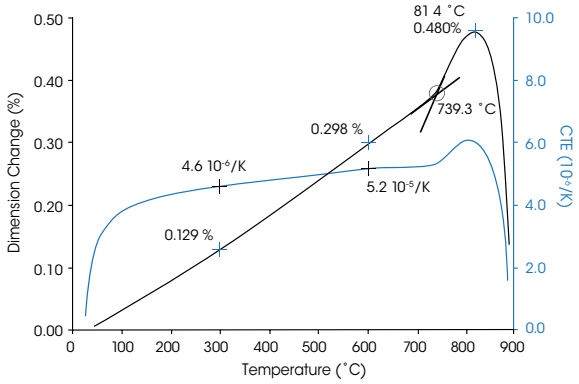


dilatometer

APPLICATIONS

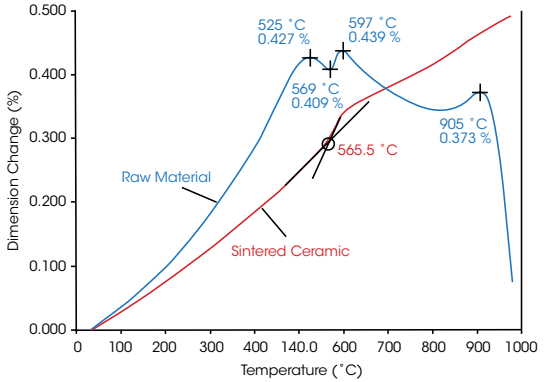
Ceramic Glazes

The coefficient of thermal expansion (CTE) is an important consideration in choosing the proper glaze for a ceramic material. If the CTE of the glaze is higher than that of the base ceramic, it will cause tension in the ceramic body upon cooling, resulting in a network of cracks and a weaker finished product. Ideally, the CTE of the glazing material should be slightly lower than that of the ceramic body which will result in a ceramic body under slight compression. In this experiment the glaze is heated through its glass transition temperature (T_g) to its softening point. The glass transition is exhibited as an inflection in the dimensional change. The CTE is also displayed as a function of temperature.



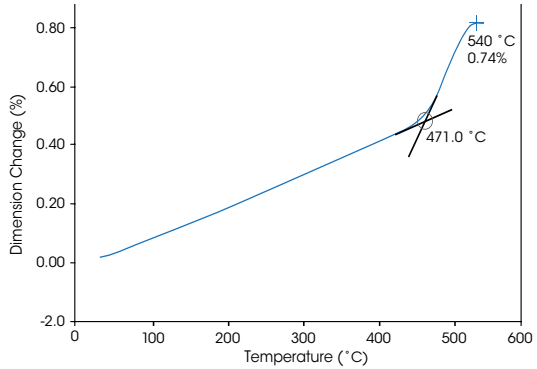
Raw vs. Sintered Ceramic

The thermal expansion behavior is shown for two samples: a fired (red) and unfired (blue) ceramic. The raw material exhibits the complex expansion and contraction behavior that is expected for a material as it undergoes both reversible (thermal expansion) and irreversible (e.g. expulsion of bound water, solid state diffusion, high temperature chemical reactions and sintering) processes. These complex behaviors are no longer present in the previously fired ceramic, leaving only thermal expansion and a phase transition at 557 °C. The ability to conduct tests in air or a controlled atmosphere allows for the direct observation of ceramic sintering processes, which are strongly influenced by the atmospheric oxygen content.



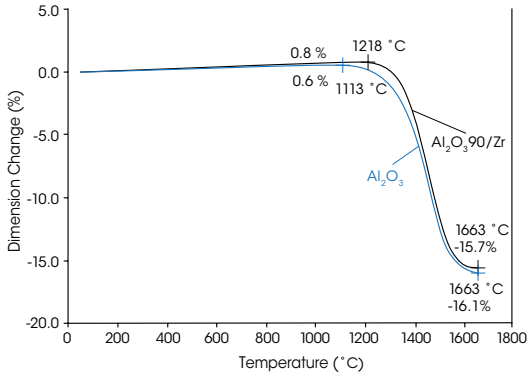
Glass Transition and Softening Temperature

Two important measurements that are often made with dilatometers are the determinations of the glass transition and the softening point. In this example the DIL 802 Differential Dilatometer measures the thermal expansion of a glass material. The sample was heated through its glass transition (T_g) at 471 °C and the test was terminated at the softening point of 540 °C. The instrument control software allows for automatic softening point detection and test abortion. This allows an unknown material to be tested to its softening point without concern of damage to the instrument.



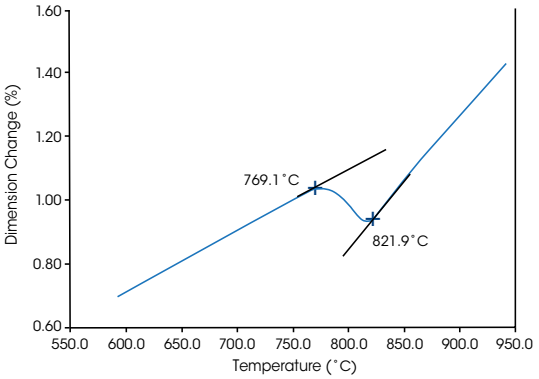
Sintering Processes

The DIL 811 vertical dilatometer is especially well-suited for the examination of rate-controlled sintering processes. In the present example, Al_2O_3 and $Al_2O_3$90/Zr are compared with respect to their thermal expansion and sintering behavior. Both materials exhibit similar thermal expansion, but the Zr alloy begins the sintering process at a much higher temperature. At the sintering temperature, both specimens were controlled with the same sintering rate criteria to termination. As seen in the figure, changes in the compositions can translate to subtle changes in their behavior, which are readily determined with the DIL 811.



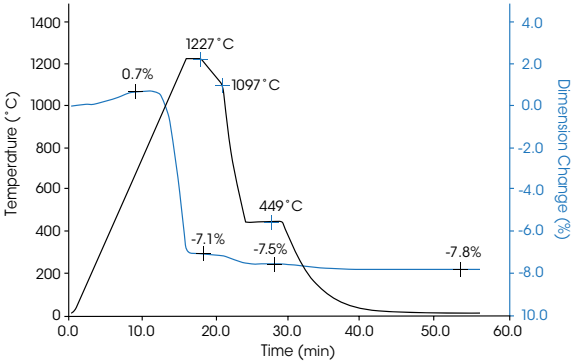
Thermal Expansion of a Thin Film

Traditionally, the measurement of a thin film in a push-rod dilatometer can be problematic due to the contact forces associated with the push-rod. The DIL 806 optical dilatometer is ideal for characterizing thin films and other materials with sample size/preparation restrictions. In this example, the thermal expansion and phase transformation of a thin steel foil is characterized by the DIL 806 non-contact optical dilatometer. The measurement process is both absolute and non-contact, so no system calibration curves are required. Sample holders are available to support thin films.



Fast-fired Ceramics

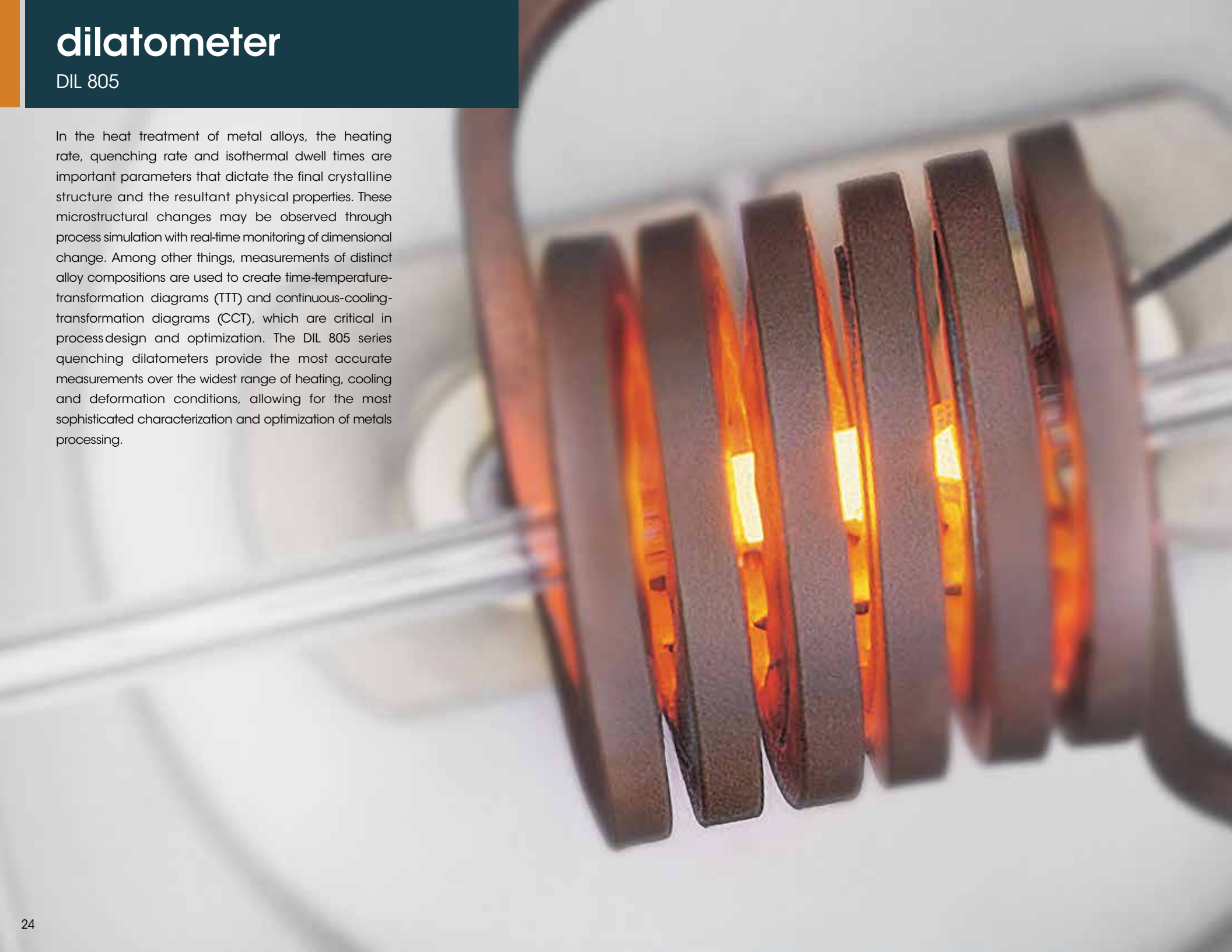
The very fast heating rates, outstanding temperature uniformity and simple programming inherent to the DIL 806 make it ideally suited to simulating industrial processes. The fast-firing process of a green body ceramic is desirable because it conserves energy and time. However, in many cases, this type of heat treatment can produce incomplete densification in the final product. In this example the sample is rapidly heated until it reaches a user-defined contraction. At this time, multiple isothermal dwells and cooling rates were used in order to closely monitor the sintering behavior of the material. By fine-tuning these temperature control parameters, based on dilatometer measurements, the industrial process can be streamlined to produce a final product with the desired physical properties and cost-advantageous processing conditions.



dilatometer

DIL 805

In the heat treatment of metal alloys, the heating rate, quenching rate and isothermal dwell times are important parameters that dictate the final crystalline structure and the resultant physical properties. These microstructural changes may be observed through process simulation with real-time monitoring of dimensional change. Among other things, measurements of distinct alloy compositions are used to create time-temperature-transformation diagrams (TTT) and continuous-cooling-transformation diagrams (CCT), which are critical in process design and optimization. The DIL 805 series quenching dilatometers provide the most accurate measurements over the widest range of heating, cooling and deformation conditions, allowing for the most sophisticated characterization and optimization of metals processing.



DIL 805L

DIL 805D

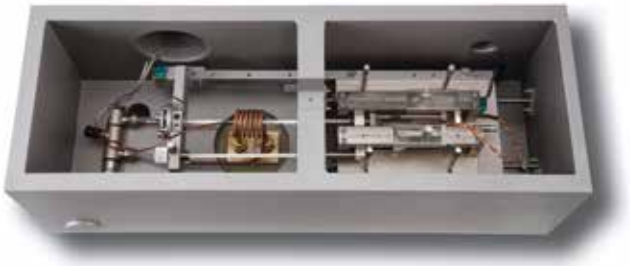
	DIL 805L	DIL 805A	DIL 805D
Temperature Range	20 °C to1500 °C	20 °C to 1500 °C	20 °C to 1500 °C
(dependent on sample material)	-150 °C to 1300 °C	-150 °C to 1300 °C	
Heating Principle	Inductive	Inductive	Inductive
Heating Rate	≤ 2000 K/s	≤ 4000 K/s	100 K/s
Cooling Rate	≤ 2500 K/s	≤ 2500 K/s	≤ 100 K/s
Sample Material and Geometry	electro-conductive solid or hollow samples OD=4 mm, L=10 mm		electro-conductive solid samples OD=5 mm, L=10 mm
Atmosphere	air, vacuum, inert gas		air, vacuum, inert gas
Resolution (ΔL/°C)	0.05 µm / 0.05 °C		0.05 µm / 0.05 °C
Deformation Force			≤ 20 kN
Deformation Rate			0.01 mm/s to 200 mm/s
Strain Rate $\dot{\varphi}$			0.001 to 20.0 s^{-1}
True Strain φ			0.05 - 1.2
Deformation			max. 7 mm
Number of deformation steps			Unlimited
Min. pause between deformation steps			40 ms

quenching dilatometer

DIL 805 ACCESSORIES

805L Quenching Dilatometer

The DIL 805L is a fully automated self-contained quenching dilatometer used to observe dimensional changes under extreme conditions of controlled heating and cooling. A solid or hollow sample is inductively heated to a temperature plateau and is then continuously cooled at a user-defined (linear or exponential) cooling rate. The phase transformation occurring in the continuous cooling process or in the isothermal dwell (which may also be a multistep transition) is indicated by the measured change in length. An array of cooling or isothermal curves represents a continuous-cooling-transformation (CCT) diagram or an isothermal time-temperature-transformation (TTT) diagram, respectively. The beginning and the end of the transformation indicate the alloy phase boundaries, e.g. ferrite, carbide, graphite, pearlite, bainite, martensite, or other eutectoid phase batches. Test experiments are flexible and may be constructed to mimic a process of any length or complexity.



805A Quenching Dilatometer

The 805A Quenching Dilatometer is the new benchmark technology for determining the dimensional changes and phase transformations of steel alloys that require the most stringent of temperature controls. Operating from -160 °C to 1500 °C (2 temperature configurations) with heating rates of up to 4000 K/s and cooling rates in excess of 2500 K/s, tests can be conducted to closely simulate the material response for any production or heat treatment process. It is designed to accommodate many different add-on modules, including the 805D Deformation adaptor, 805T Tension adaptor, and the DTA/DSC measuring head. This instrument is a powerful and versatile tool for the determination of critical parameters in steel manufacturing and heat treatment processes.

Sub-zero Module

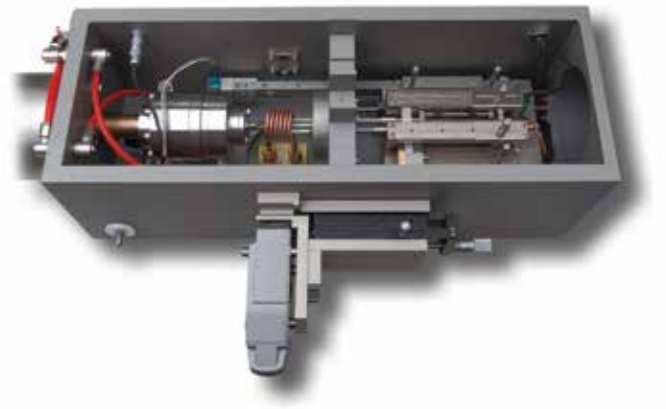
In many cases the martensitic finish temperature, Mf, of a steel lies well below room temperature. This add-on module operates from -160 °C to 1300 °C, with attainable controlled quenching rates in excess of 2500 K/s, and allows for the complete characterization of the austenite to martensite transformation. This unique quenching technology passes helium gas through a copper heat exchanger submersed in a liquid nitrogen bath before delivery to a hollow sample. The design greatly improves heat transfer by eliminating many of the issues associated with liquid nitrogen cooling, including condensation, material interaction, and imprecise response rates.

805D Deformation Dilatometer

Steel processes, such as hot or cold rolling, require detailed knowledge of the time-temperature-transformation diagram after deformation (DTTT diagram). With the deformation module, the principle of the 805A quenching dilatometer is extended to include controlled deformation. Solid samples are compressed using various deformation programs (e.g. linear, multi-level with a constant deformation force or rate) with controlled forces up to 25 kN or rates up to 200 mm/s. An unlimited number of deformation steps can be performed with a pause between steps of only 40 ms. This unique technology enables the control of cooling and deformation processes in order to create a DTTT diagram. The 805D is also used to examine creep and relaxation processes.

805T Tension and Compression Adapter

The 805T extension further extends the capabilities of the instrument to alternating tensile and compressive loading. The expansion of a clamped sample is measured during heating or cooling to emulate mill processing. Once the desired temperature is achieved, it is held isothermally while the desired mechanical cycling is performed. Force-controlled or strain-controlled cycles are available up to 8 kN or 20 mm/s, respectively. Additionally, tensile loading to fracture lends additional information about the final performance characteristics of the material. These data are used to generate true-stress vs. true-strain or stress/strain cycling plots.

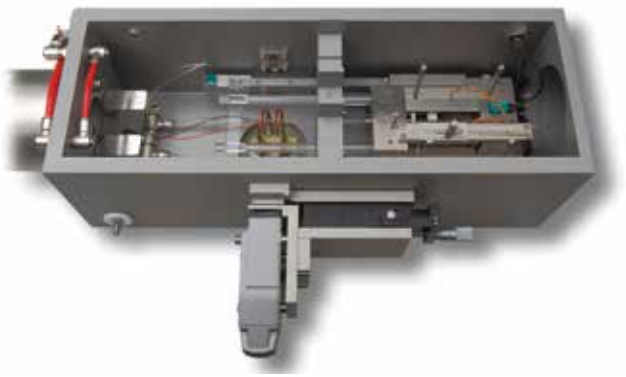


quenching dilatometer

DIL 805 ACCESSORIES

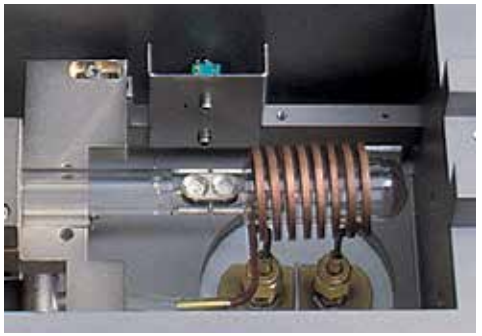
Alpha Measuring System

The Alpha measuring head uses low-expansion fused silica components in conjunction with a true differential LVDT for high-precision expansion measurements. This system allows the DIL 805 to be used for traditional push-rod dilatometer studies such as the determination of the coefficient of thermal expansion (CTE) and the softening point.



DTA/DSC Measuring Head

The DTA/DSC measuring head is custom-designed for the analysis of phase transformation and precipitation processes in metals. With heating and cooling rates of up to 500 K/min, an identical temperature program (as in many of the quenching studies) can be used in conjunction with a traditional thermal analysis measurement.



Induction Heating Coil

The custom-designed induction heating coil allows for rapid inductive heating at rates up to 4000 K/s of an electrically conductive solid or hollow sample. During a test, only the sample is heated so there is no associated furnace/insulation cool-down period, and another sample can be loaded immediately upon test completion. The hollow-core inner coil also serves as the purge gas conduit focused at the heating zone, ensuring an inert environment throughout the test. Specially designed heating rings are also available for use with samples that are not electrically conductive.

Optical Module

Traditional dilatometers measure the thermal expansion of a material in one axial direction and have an inherent drift associated with the thermal interaction at the contact point between the push-rod and the sample, especially during isothermal dwells. With the optical expansion module, contraction/expansion is monitored in two directions during the test run. The measurement is non-contact and absolute, so it is free from interaction with dilatometer temperature gradients and a calibration correction is not necessary. The optical module is available for the quenching, deformation and tension/compression configurations, and produces results that are unachievable by conventional methods.

Thermocouple Placement Device

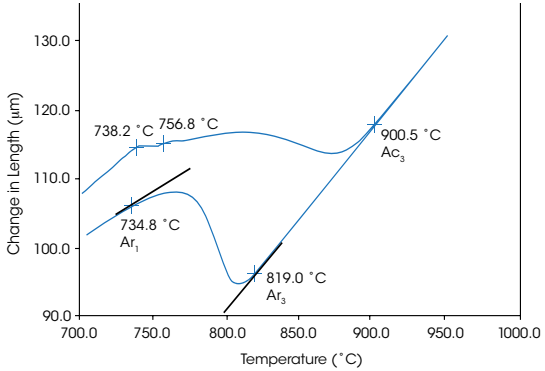
Precise temperature control requires temperature monitoring in close proximity to the sample. The easy-to-use thermocouple placement device reproducibly spot welds up to 3 thermocouples directly onto the sample for temperature resolutions of 0.05 °C across the full temperature range. The welding current and time, the contact pressure, and the inert gas purge can be adjusted to ensure a strong spot weld onto the sample.

dilatometer

APPLICATIONS

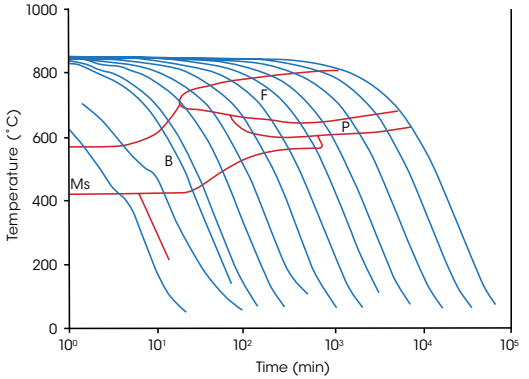
Steel Phase Transformation

Phase transformations in steel are highly path dependent, reflecting the effects of earlier processing steps on subsequent phase composition. The transitions between different phases of steel are especially clear when measured by the DIL 805A Quenching Dilatometer, and the temperatures at which they occur are critical in the construction of the TTT and CCT diagrams. In this example, the first ramp rate heats the sample above its austenitic temperature, at which time it is quenched. The plot shows the start (Ar_3) and finish (Ar_1) of the phase transformation from austenite to ferrite. These two temperature points can then be fitted to a CCT diagram based on the quench rate.



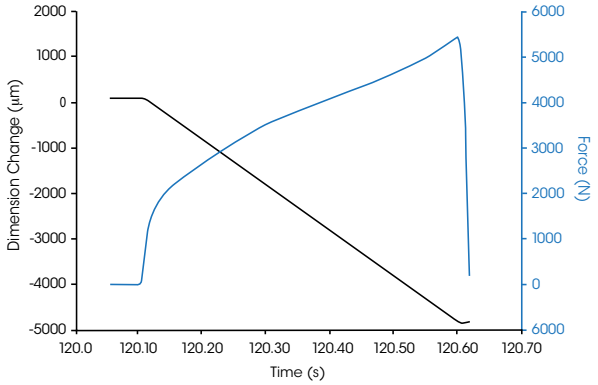
Continuous Cooling Transformation Diagram

As the name suggests, the CCT phase diagram represents the phase transformation of a material when it is cooled at various controlled rates. In the heat treatment of steel, the CCT diagram is used to predict the final crystalline structure of the processed steel. This crystalline structure determines the physical properties and suitabilty for the application in which the material will be used. The DIL 805A is the ideal tool to observe small dimensional changes under extreme conditions of controlled cooling. Software is available for the seamless preparation of TTT or CCT diagrams.



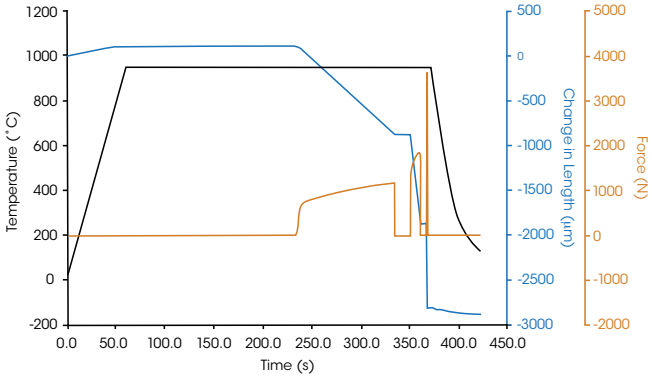
Force Deformation

The 805D add-on module can precisely control the strain-rate of a sample and measure the resultant force required to achieve this. In this high speed test run, a deformation rate of 10 mm/s is used for a maximum displacement of 5 mm (Strain 0.50). The force exerted by the hydraulic ram is closely monitored, and both data sets can be used to plot the true stress vs. true strain curve of the material.



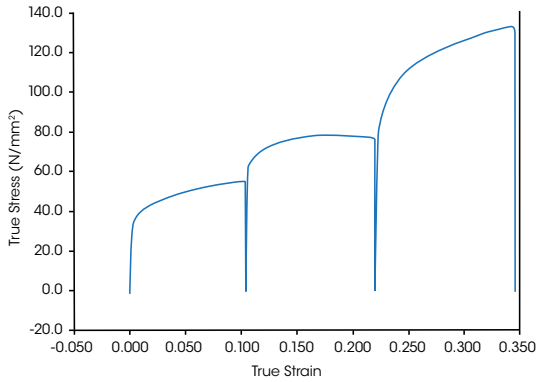
3-Step Deformation Test

Simulating metal processing techniques, and the phase transformations that take place upon quenching or heat treating, are important measurements to perform in order to accurately control the crystalline structure and its inherent physical properties. The DIL 805A/D is the ideal instrument for optimizing the quench rate after these multi-step deformations. In this example, after the initial heating and resultant thermal expansion, the parcel of steel is held isothermally and goes through a series of 3 deformation steps: an initial 1mm deformation over a 100 s time period; a second 1 mm deformation over a 10 s time period, and finally a seemingly instantaneous force applied for the final 1 mm deformation. After another 10 s dwell at the isothermal processing temperature, the material is quenched and the contraction and phase transformation is measured. Using this measured data, the manufacturer can streamline their processing for repeatable production of steel with the desired physical properties.



True Stress vs. True Strain Curves

This plot is the true stress vs. true strain curves, measured during the deformation steps in the above example. Please note that the "instantaneous" force pulse in the third deformation step was measured and can now be analyzed. With over 100,000 data points taken per second, the DIL 805A/D is a powerful tool that can further help engineers develop the mechanical aspects of the processing line.



notes

tainstruments.com

L90016.000