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
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Elstree, United Kingdom
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Milano, Italy
Warsaw, Poland
Prague, Czech Republic
Sollentuna, Sweden
Helsinki, Finland
Copenhagen, Denmark
Chicago, IL USA
São Paulo, Brazil
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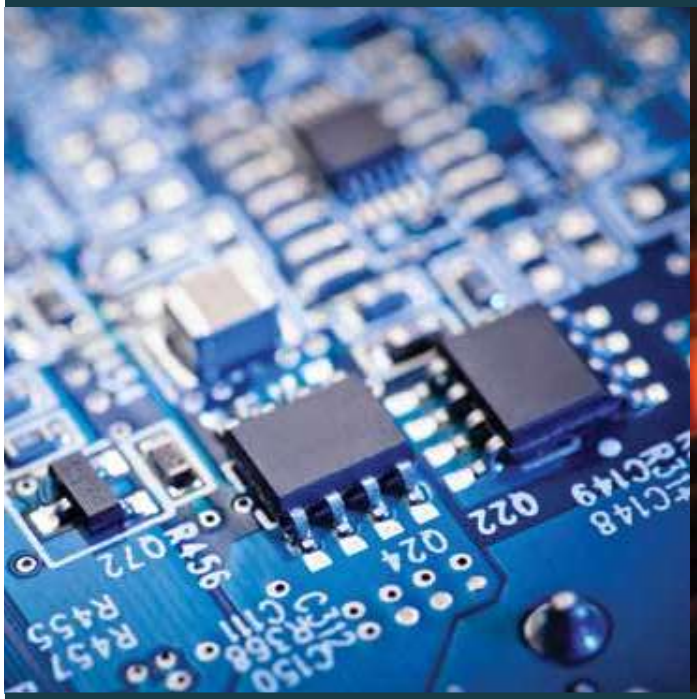
Montreal, Canada





dilatometry

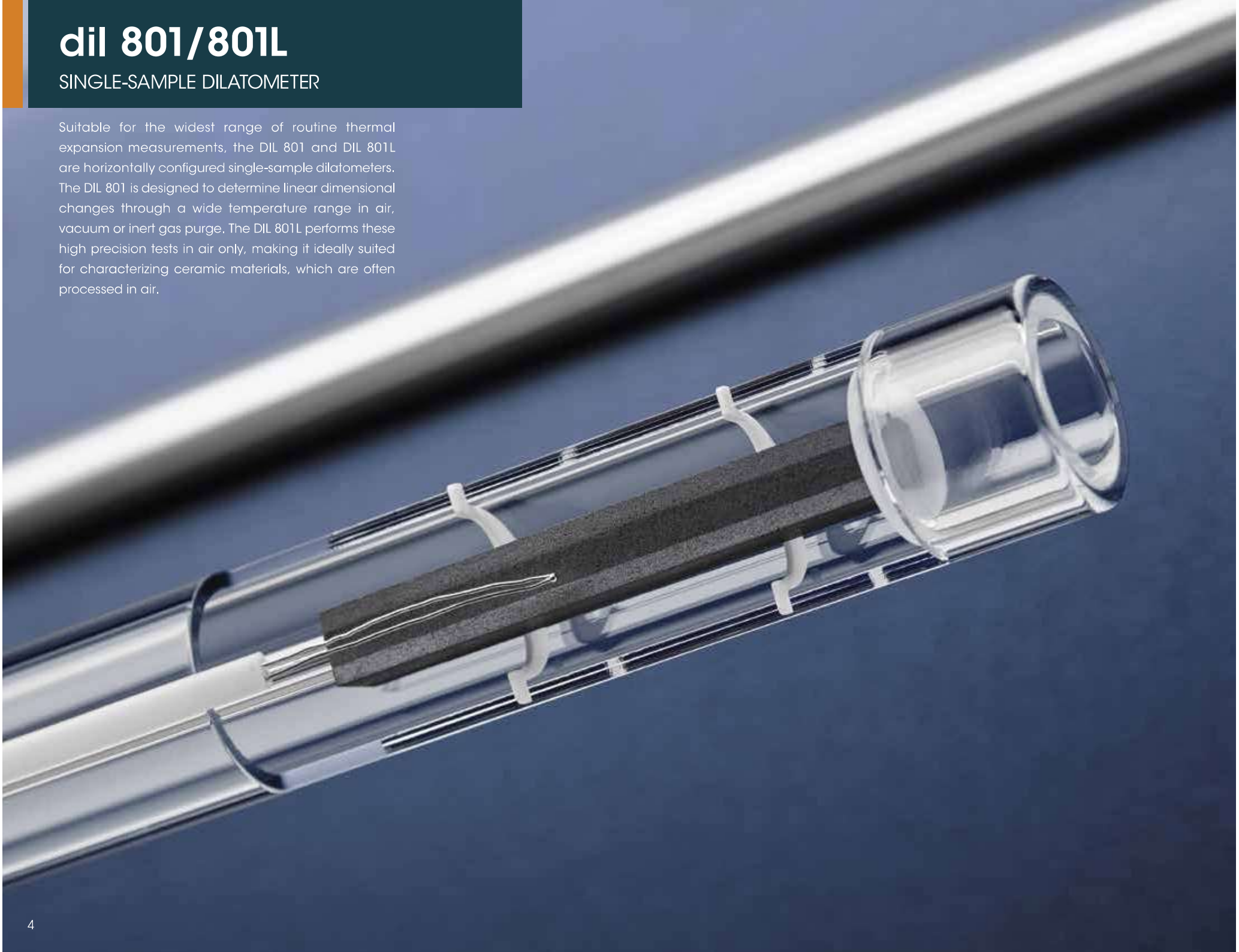
Every TA Instruments dilatometer precisely measures dimensional changes of a specimen brought about by changes in its thermal environment. Typical measurements include thermal expansion, annealing studies, determination of phase transitions and the glass transition, softening points, kinetics studies, 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

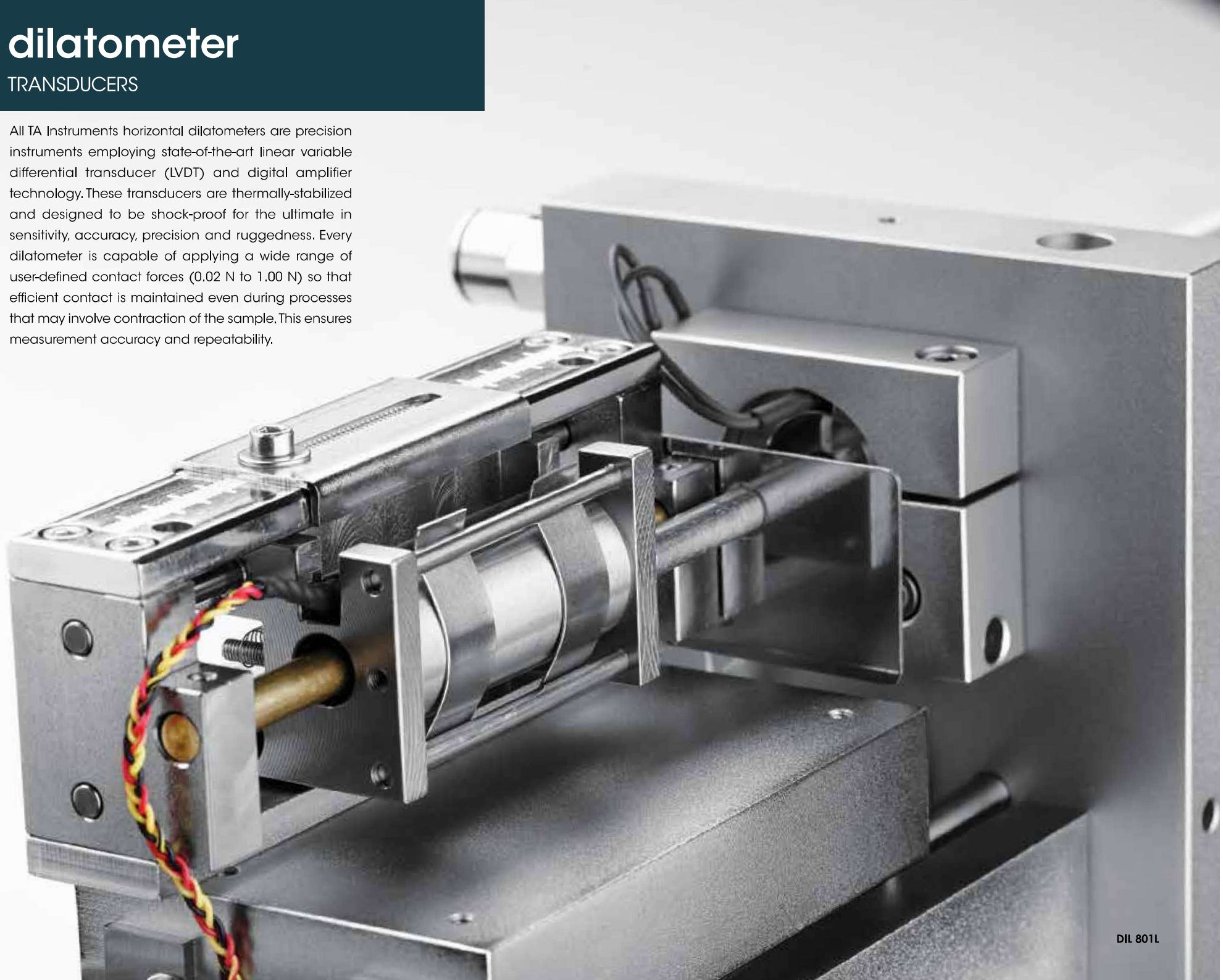
To maximize sample throughput, the DIL 803/803L offers dual-sample simultaneous operation in a horizontal dilatometer. The DIL 803/803L can also be operated as a differential system, using an inert reference specimen to reduce the influence of system expansion, increasing accuracy under dynamic temperature 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

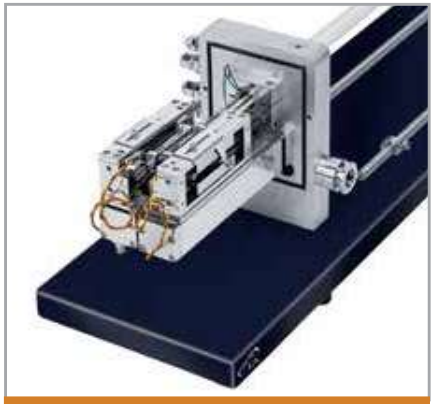
All TA Instruments horizontal dilatometers are precision instruments employing state-of-the-art linear variable differential transducer (LVDT) and digital amplifier technology. These transducers are thermally-stabilized and designed to be shock-proof for the ultimate in sensitivity, accuracy, precision 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.



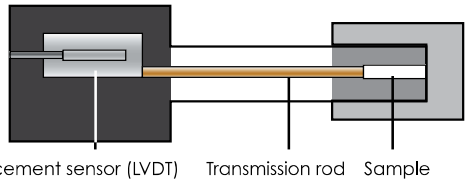
DIL 801L

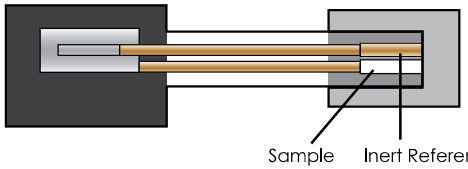
DIL 801 (Standard)

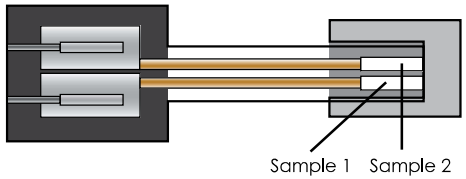
DIL 802 (Differential)



DIL 803 (Dual-Sample)



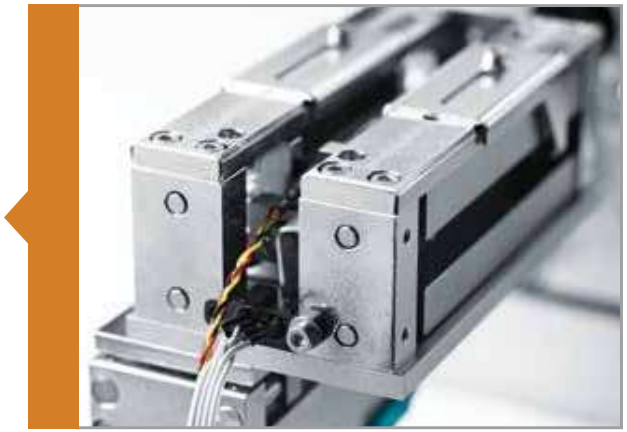




True Differential Measurement DIL 802

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.

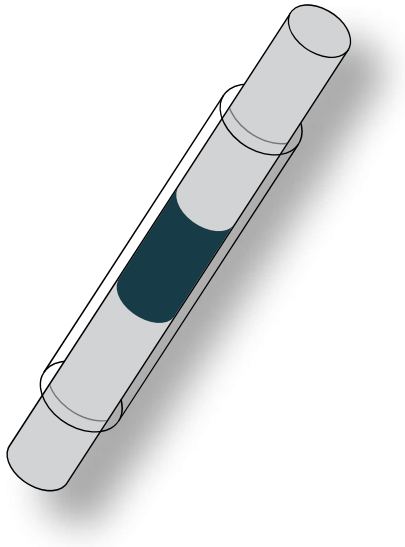
Testing Standards

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.

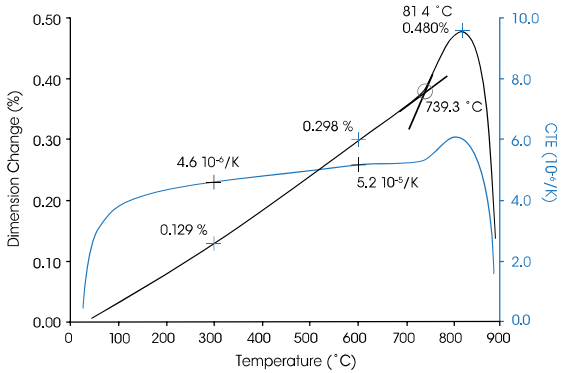


dilatometer

APPLICATIONS

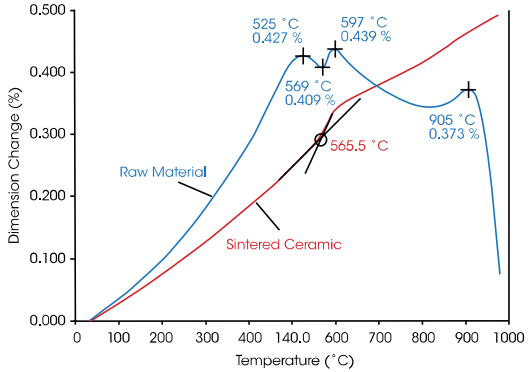
Ceramic Glazes

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.



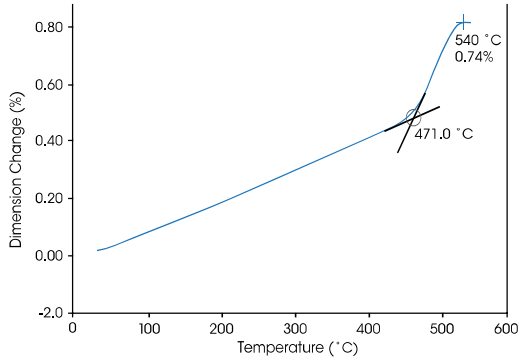
Raw vs. Sintered Ceramic

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.



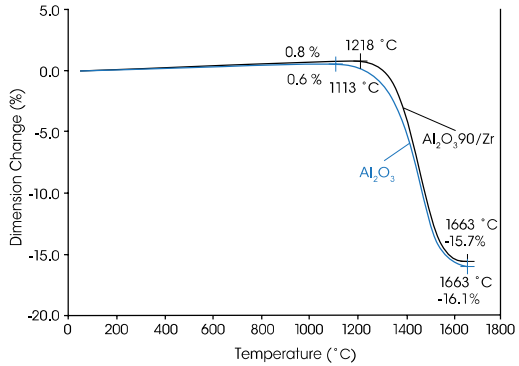
Glass Transition and Softening Temperature

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.



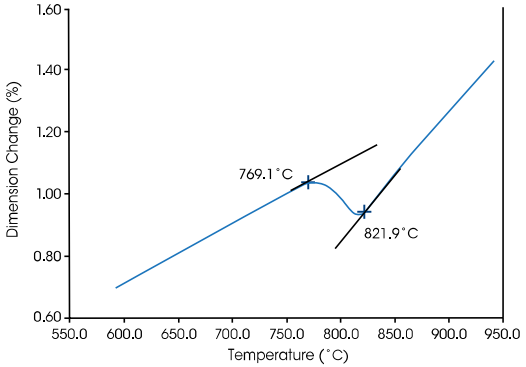
Sintering Processes

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.



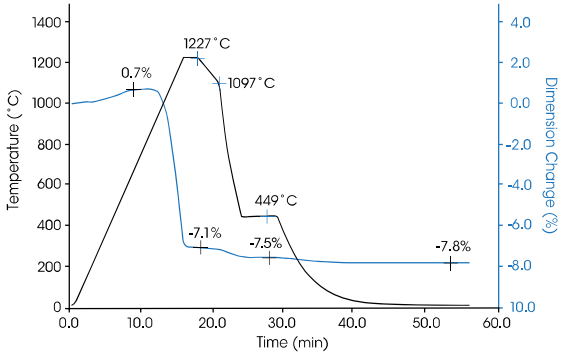
Thermal Expansion of a Thin Film

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.



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