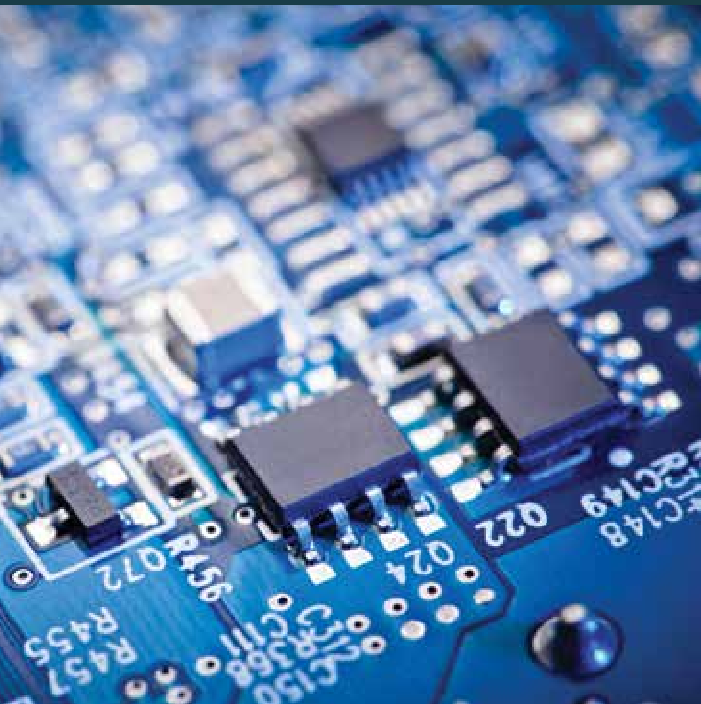
TA Instruments

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.

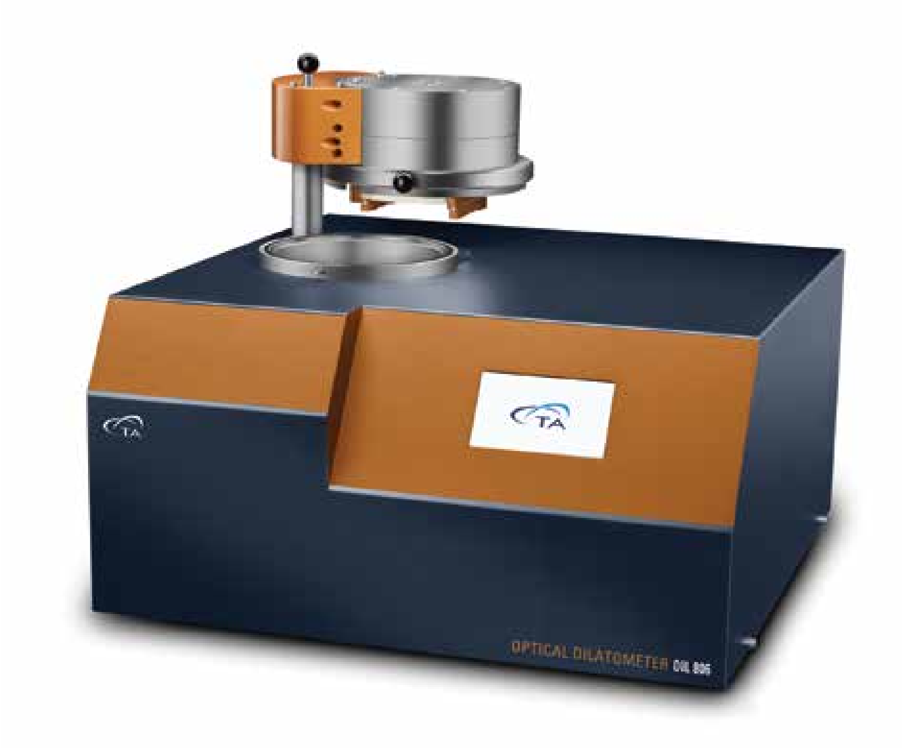
Each application of dilatometry has its own experimental requirements. That is why TA Instruments provides dilatometers in four basic types, each of which have flexibility of sample atmosphere, temperature and measurement control. Only TA Instruments can provide the right instrument to match your needs—no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

dil 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	$0.05 \times 10^{-6} K^{-1}$
Temperature Range	-150 °C to 600 °C RT to 900 °C RT to 1400 °C
Atmosphere	vacuum, inert gas, air DIL 806L: air only

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

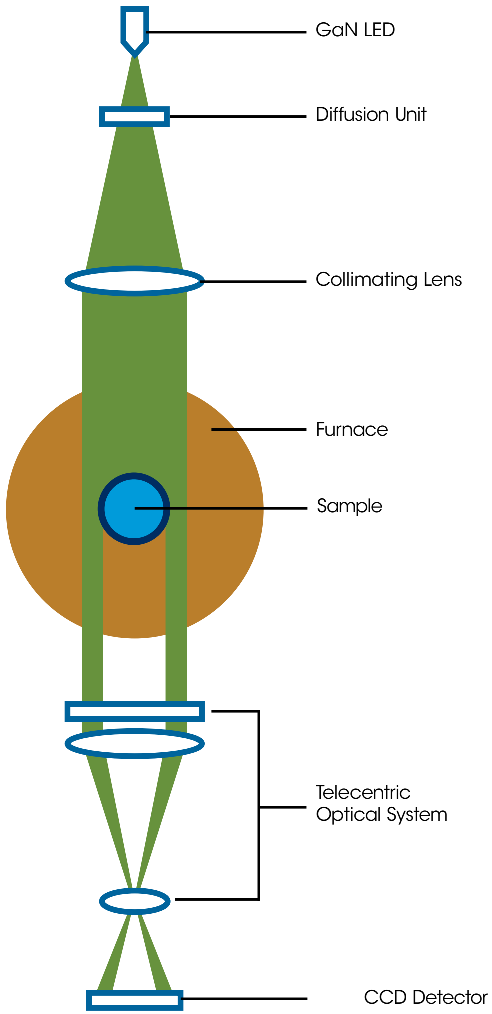
The measurement of the DIL 806 is an intrinsically absolute measurement, unaffected by system thermal expansion that accompanies the programmed changes in temperature. Only the sample is subjected to temperature excursions; both the light source and detector are well-isolated from these changes. Consequently, 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.

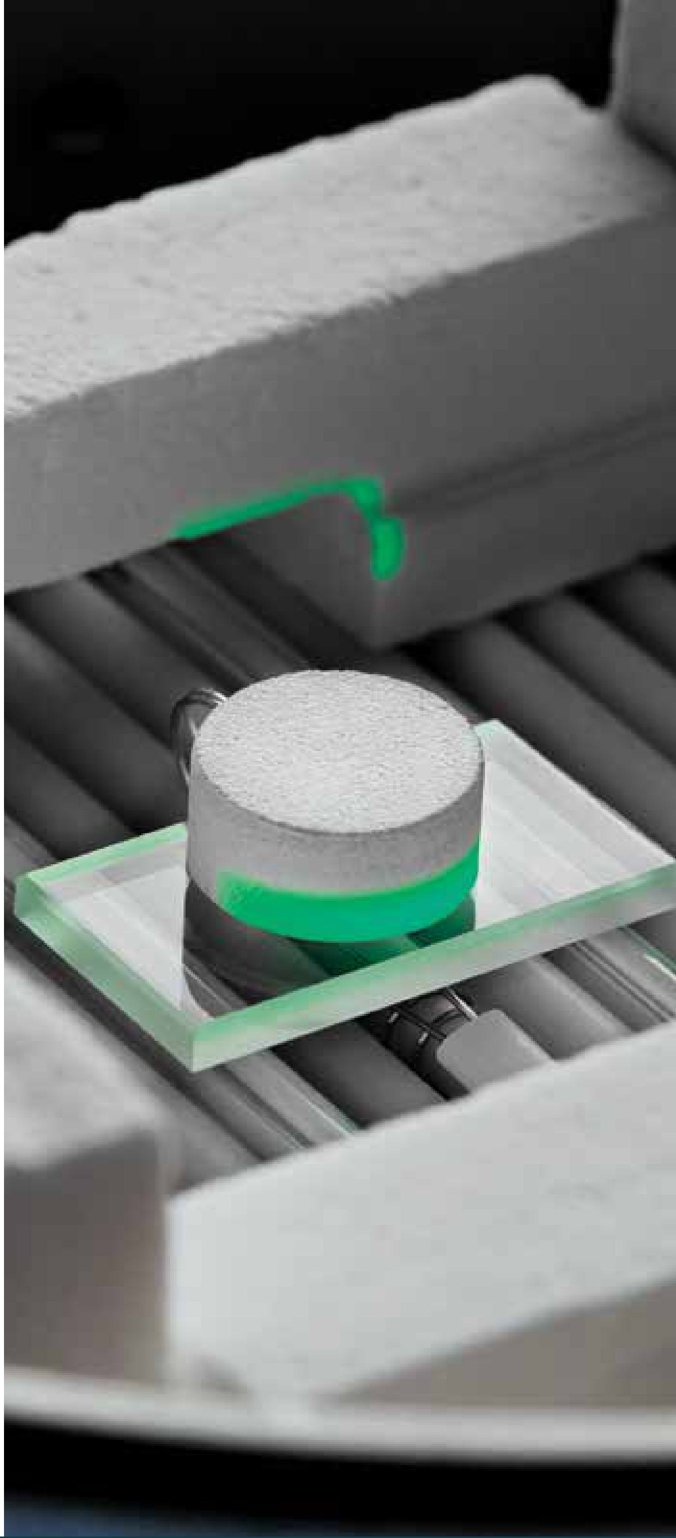
The absence of physical contact with the measuring system further enhances temperature uniformity. Push-rods can act as heat sinks, generating hot or cold spots at the point of contact with the sample. The DIL 806 is free of these contact points, ensuring that the entirety of the sample is at a uniform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

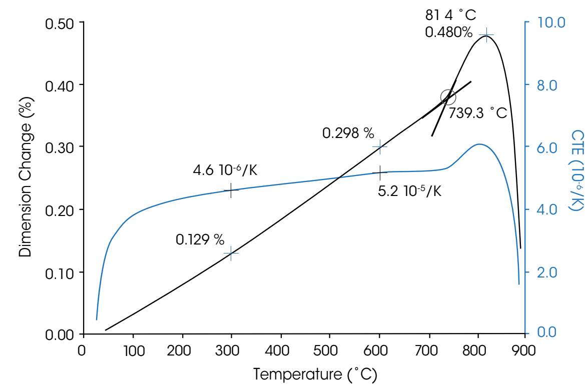
Sample positioning is greatly simplified by the wide measurement area. Using a measurement area 30 mm wide, the instrument works equally well with a sample positioned anywhere in this range. This simplifies sample loading by removing strict restrictions on sample position.

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.

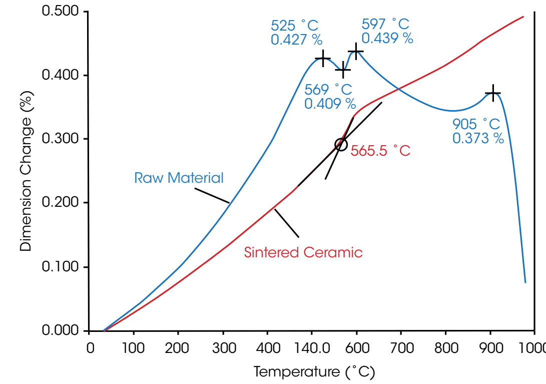
Because the DIL 806 can accommodate many sample types and shapes, it is a natural complement to other measurements. The same specimen may be measured in the DIL 806 as is measured by dynamic mechanical analysis, the flash technique for thermal conductivity, surface hardness density and more.





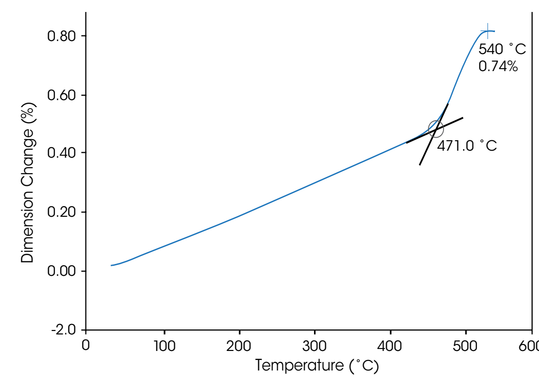
Ceramic Glazes

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.

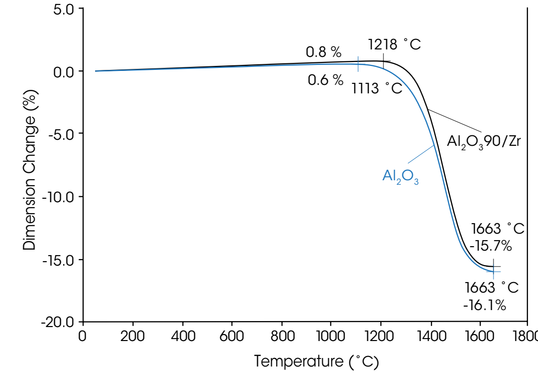


Glass Transition and Softening Temperature

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.

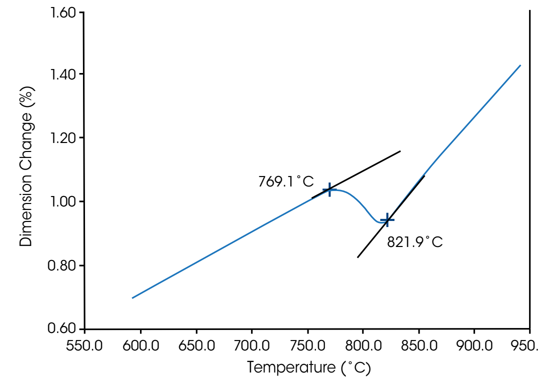
Sintering Processes

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.



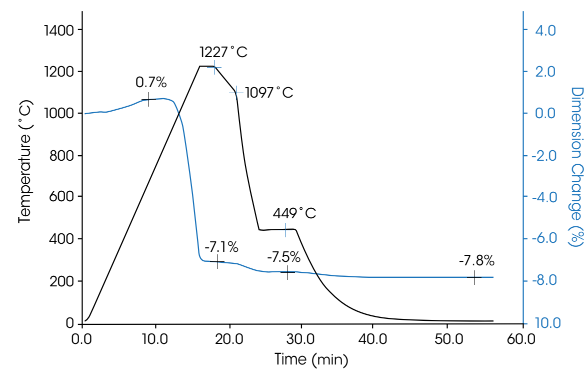
Thermal Expansion of a Thin Film

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.



tainstruments.com

L90016.000