Dilatometry
Method, Instruments, Applications – From -180°C to 2800°C
Dilatometry is a thermoanalytical technique used to measure the expansion or shrinkage of solids, powders, pastes and liquids under negligible load when subjected to a controlled temperature/time program.

A precise understanding of this behavior can provide insight into firing processes, the influence of additives and raw materials, densification and sintering properties, reaction kinetics, phase transitions, and thermal shock. In addition, it can be used for glaze development and to match CTEs (glaze-ceramic, metal-ceramic in the automotive industry).

Dilatometry can be applied not only to solid samples, but also to powders, pastes, and even liquids.

It can also be used to carry out rate-controlled sintering studies on reactive powders in fields such as advanced ceramics or powder metallurgy.

Due to large technological advances, this thermal analysis method can now measure even the slightest of thermal behaviors in ceramics.

Recent innovations in dilatometer hardware and software design promise to augment the possibilities provided by thermal expansion measurement. Such innovations include the application of new thermokinetic analysis techniques to model- and construct-optimized firing and sintering processes, the combination of dilatometry with calculated Differential Thermal Analysis (c-DTA®) for a better understanding of thermal behavior, and the designing of new hardware to improve low-temperature thermal expansion measurements.

All NETZSCH dilatometers are designed in accordance with standards such as DIN EN 821, DIN 51045, ASTM E831, and ASTM E228.

DIL 402 C as an example of a pushrod dilatometer.
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Dilatometry

Phase Transition
Refractories
Building Materials
Sintering
Alloys
Density
Metals
Shrinkage
Softening
Ceramics
Porcelain
Expansion
Polymers
Composites
Glass
CTE

3
Dilatometry – Method and Principle of Operation

Method and Principle of Operation

Method

Pushrod dilatometry is a method for determining dimensional changes versus temperature or time while the sample undergoes a controlled temperature program. The degree of expansion divided by the change in temperature is called the material’s coefficient of expansion ($\alpha$) and generally varies with temperature.

Principle of Operation

To perform a dilatometric analysis, a sample is inserted into a special holder within a movable furnace. A pushrod is positioned directly against the sample and transmits the length change to a linear variable displacement transducer (LVDT).

As the sample length changes during the temperature program, the LVDT core is moved, and an output signal proportional to the displacement is recorded. The temperature program is controlled using a thermocouple located either next to the heating element of the furnace or next to the sample.

Since the sample holder and the front part of the pushrod are being exposed to the same temperature program as the sample, they are also expanding. The resulting dilatometer signal is therefore the sum of the length changes of sample, sample holder, and pushrod.

It is thus necessary to correct the raw dilatometer data in order to obtain a true view of sample behavior. There are two correction methods: the application of tabulated expansion data, or – often more precise – of a correction curve to eliminate systematic error.

\[
\alpha = \frac{1}{L_0} \left( \frac{\Delta l}{\Delta T} \right)
\]

$\alpha$  coefficient of expansion  
$L_0$  initial sample length  
$\Delta T$  change in temperature  
$\Delta l$  change in length  

Schematic of a pushrod dilatometer
Results and Measuring Accuracy

The thermal expansion in percent is an important value for evaluating a material’s performance.

In this diagram, the length changes of 3 test runs (lines) on polycrystalline alumina (Al₂O₃) are presented together with the corresponding data from literature (crosses).

The differences between these values at 500°C, 1000°C, and 1500°C generally amount to less than 1%, demonstrating the outstanding accuracy of the NETZSCH DIL 402 PC which was used for these measurements.

Advantage of a Horizontal Pushrod Dilatometer

The superior thermal uniformity of a horizontal pushrod dilatometer can be attributed to very low temperature gradients. A horizontal furnace is not subject to convection parallel with the sample.

Particularly for long samples, a horizontal dilatometer system is essential in ensuring that the temperature distribution is reasonably uniform.

Additional Advantages

- Wide temperature range
- Easy handling
- High accuracy
- Homogeneous temperature profile
- Flexible sample geometry
- Low risk of contamination (LVDT)

DIL Measurement Information

- Linear thermal expansion
- Coefficient of thermal expansion (CTE)
- Volumetric expansion
- Shrinkage steps
- Glass transition temperature
- Phase transitions
- Sintering temperature/sintering step
- Density change
- Softening points
- Influence of additives/raw materials
- Decomposition temperature – e.g., of organic binders
- Anisotropic behavior
- Optimizing of the firing process
- Rate-controlled sintering (RCS)
- Caloric effects by using c-DTA®
- Thermokinetics
The DIL 402 PC is specially tailored to the needs of the ceramic and glass industry. High resolution and stability, a wide measurement range, and a robust and compact design are only some of the many advantages of this cost-effective instrument. This dilatometer combines easy operation, high adaptability to different applications, and outstanding performance in one instrument. The optimized design of the measurement system with inductive transducer compensates for temperature fluctuations and yields highly reproducible data. A chiller is not required.

The DIL 402 PC operates in accordance with nearly all national and international standards (e.g., DIN 51045, or JIS R 2207-contact version).

Design

The instrument has a horizontal design with an easy-to-operate furnace. A large recess in the tube-type sample carrier facilitates the placement of samples even when their geometries are less than ideal. A thermocouple in direct proximity to the sample ensures reproducible temperature measurement.

Furnaces

The interchangeable furnaces with temperature ranges up to 1200°C and 1600°C allow for the analysis of expansion control in solids, green bodies, powders and pastes, for production and quality assurance purposes across a variety of applications.

Sample Holder Systems and Sample Dimensions

Exchangeable sample holder systems made of alumina and fused silica are available. The maximum sample dimensions are 50 mm in length with a diameter of up to 12 mm (optionally 19 mm).

The forced-air cooling system of the 1600°C furnace makes it possible to cool from maximum sample temperature down to room temperature.
At a Glance – The Characteristics of the DIL 402 PC

- High flexibility with two interchangeable furnaces
- Various accessories for special applications (sample holders and containers)
- Large sample dimensions
- Ease of operation
- Highly reproducible results

### Technical Key Data for the DIL 402 PC

<table>
<thead>
<tr>
<th>Feature</th>
<th>Details</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature range/furnaces</td>
<td>RT to 1200°C / RT to 1600°C</td>
</tr>
<tr>
<td>Heating rate</td>
<td>0.1 to 50 K/min</td>
</tr>
<tr>
<td>Temperature precision</td>
<td>0.1 K</td>
</tr>
<tr>
<td>Measuring range</td>
<td>500/5000 µm</td>
</tr>
<tr>
<td>Load at the sample</td>
<td>15 cN to 45 cN (optionally 45 cN to 100 cN)</td>
</tr>
<tr>
<td>Δl resolution</td>
<td>8 nm/digit</td>
</tr>
<tr>
<td>Sample diameter</td>
<td>Up to 12 mm (optionally 19 mm)</td>
</tr>
<tr>
<td>Sample length</td>
<td>Up to 25 mm / Up to 50 mm</td>
</tr>
<tr>
<td>Sample holders</td>
<td>Alumina / Fused silica</td>
</tr>
<tr>
<td>Special sample holder systems</td>
<td>3-point bending mode (determination of the firing stability, set made of Al₂O₃) / Fibers in tension / Thin metals and ceramic foils / Penetration</td>
</tr>
<tr>
<td>Sample containers</td>
<td>Powders / Pastes / Metals and glasses in the liquid state</td>
</tr>
<tr>
<td>Atmosphere</td>
<td>Oxidizing static and dynamic / Inert dynamic</td>
</tr>
<tr>
<td>c-DTA®</td>
<td>For temperature calibration and detection of endo- and exothermal effects</td>
</tr>
<tr>
<td>Gas flow controller</td>
<td>For purge gas (optional)</td>
</tr>
</tbody>
</table>

Special sample holder for the determination of the firing stability
The Dilatometer DIL 402 C features an excellent set of capabilities optimized to fulfill any potentially occurring application need. These include flexibility of temperature range, sample holder, and gas atmosphere, along with high accuracy and easy handling – the result of over 50 years of experience in building dilatometers.

The state-of-the-art design is compact and extremely flexible; it uses a number of interchangeable furnaces with horizontal movement and provides easy access to the sample holder. It is the perfect instrument for applications in the areas of not only traditional ceramics and glass, but also advanced ceramics and powder metallurgy (e.g., when carrying out sintering studies on reactive powders).

Measurement System
The high-resolution displacement transducer is employed in a Vacodil® (Invar) measuring system. The maximum measuring range is 5000 μm. The extremely low drift exhibited by the system guarantees measurements with very high repeatability, accuracy and long-term stability for application temperatures up to 2000°C.

Wide Temperature Range and Exchangeable Furnaces
Six easily exchangeable furnaces are available to cover a temperature range of -180°C to 2000°C. This not only provides flexibility for different applications, but also ensures the smallest temperature gradient along the sample for both low- and high-temperature applications.

Motorized Pushrod and Automatic Zeroing
The motorized pushrod and the easy-to-move furnace make it quite simple to insert samples, even when sample geometries are less than ideal. The pushrod is brought into contact with the sample via a software command which also automatically brings the LVDT to a zero/central position. Should a different starting position be required for large expansion or shrinkage, this can be accomplished with a simple software command as well.
Atmosphere – High Vacuum

The vacuum-tight design of the DIL 402 C allows for careful control of the atmosphere and pure gas conditions. For example, the system can be evacuated and back-filled with pure inert or non-reactive gas. Static or dynamic reactive gas atmospheres can be applied as well. Materials sensitive to oxygen can be studied under pure inert gas conditions. To provide such conditions, an evacuation system (optionally available) with a two-stage rotary pump can be hooked up to the standard vacuum flange. For high-vacuum requirements (10⁻⁴ mbar), a turbo molecular pump system is available.

Adjustable Contact Pressure

In the DIL 402 C, the contact force of the pushrod can be adjusted between 15 cN and 45 cN, and optionally between 45 cN and 100 cN. An almost frictionless ball-bearing design supporting the pushrod guarantees smooth detection of changes in sample dimension.

Thermostatic Control

To prevent any thermal influence from the furnace or from changes in room temperature, the LVDT system is kept at a constant temperature using a precise thermostat. This guarantees reproducible measurements even in the most sensitive measuring range.

Control System

The thermal analysis system controller, TASC 414, combines a multi-step programmer and PID controller with a high-resolution data acquisition system. A Sample Temperature Controller (STC) unit provides for excellent sample temperature control.

Evolved Gas Analysis

The vacuum-tight design of the DIL 402 C is ideally suited for connection to a mass spectrometer via a capillary coupling or to an FT-IR via a transfer line. The outgassing of additives, organic binders and decomposition products can thus be studied.
Large Sample Size

The maximum sample diameter for the standard sample holders is 12 mm. Special fused silica, alumina and graphite sample holders are available for diameters up to 19 mm.

Tube-Type Sample Carrier

The tube-type sample carrier is standard for the DIL 402 C. The sample is placed within the tube. Supports can be used for centering and to prevent direct contact (sticking) between the sample and carrier. The sample carriers are available in fused silica (max. 1100°C), alumina (max. 1680°C), or graphite (max. 2000°C).

Rod-Type Sample Carrier

The fused silica or alumina tube-type sample carriers can optionally be replaced with rod-type sample carriers for ideal heat transfer to the sample. These consist of three support rods, leaving the sample open to the heat source and the gas atmosphere. This results in an increase in accuracy.

Sample Containers

For measurements on pastes, powders, molten metals and other material configurations, special sample containers made of fused silica, alumina, sapphire or graphite are optionally available.
## Technical Key Data for the DIL 402 C

<table>
<thead>
<tr>
<th>Category</th>
<th>Details</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Temperature range</strong></td>
<td>-180°C to 2000°C</td>
</tr>
</tbody>
</table>
| **User-interchangeable furnaces** | -180°C to 500°C (two versions available; closed or open to one side)  
  Stainless steel: -150°C to 1000°C (with extended heating zone)  
  Tube furnace: RT to 1000°C (with protective tube made of fused silica)  
  SiC: RT to 1600°C  
  Rh: RT to 1600°C (especially for use in glove box)*  
  Graphite: RT to 2000°C |
| **Interchangeable protective tubes** | Depending on furnace, available as open or closed tubes; made of:  
  Alumina  
  Fused silica  
  Glassy carbon |
| **Heating rate**                 | 0.01 to 50 K/min |
| **Temperature precision**        | 0.1 K |
| **Cooling systems**              | Liquid nitrogen (low-temperature furnaces)  
  Forced air integrated in the SiC furnace and tube furnace  
  Water cooling (graphite furnace) |
| **Atmosphere**                   | Inert  
  Oxidizing (graphite furnace up to 1680°C with special protective tube)  
  Reducing  
  Vacuum (<10⁻⁴ mbar (10⁻² Pa))  
  Static and dynamic operation |
| **Gas flow control options**     | Gas flow control supplement for 1 gas path  
  Mass flow controller (MFC), box for 3 gases |
| **c-DTA®**                       | For temperature calibration and detection of endo- and exothermal effects |
| **Measuring range**              | 500/5000 μm |
| **Load at the sample**           | Standard: 15 cN to 45 cN  
  Optionally: 45 cN to 100 cN |
| **Δl resolution**                | 0.125 nm/digit, 1.25 nm/digit |
| **Sample diameter**              | Up to 12 mm (optionally 19 mm) |
| **Sample length**                | Up to 25 mm (standard)  
  Up to 50 mm (optionally for low-temperature furnaces) |
| **Interchangeable sample holders** | Tube-type: fused silica, alumina, graphite  
  Rod-type: fused silica or alumina |
| **Special sample holder systems** | Thin metal, ceramic and glass foils  
  Penetration  
  Bending (especially for determination of firing stability, set made of Al₂O₃)  
  Rod-type sample holder in combination with the humidity generator and humidity sensor |
| **Sample containers**            | For pastes, liquids, waxes and molten metals (incl. aggressive samples) in fused silica, alumina, sapphire, graphite and steel |
| **Humidity generator**           | Optional for measurements under a relative humidity of between 5% and 90%; temperature range from 30°C to 80°C |

*Special glove box designs available upon request*
DIL 402 CD – Dual and Differential Excellence

The Dual and Differential Dilatometer DIL 402 CD – Two Instruments in One

The DIL 402 CD offers the highest level of performance in thermal expansion measurements. In the double sample arrangement, the system is capable of high sample throughput. In the differential arrangement, the system offers calibration-corrected measurements in one run and improved long-term drift stability.

Design

The horizontal, vacuum-tight instrument construction offers specific advantages, especially in the dual sample arrangement: homogeneous heating of both samples, easy sample insertion, and effective protection of the measuring system by gas flow. In addition, no convective heat transfer takes place between the measuring system (two high-resolution inductive displacement transducers) and the sample area, thus eliminating increased drift and noise.

Advantages of the Double Sample Arrangement

- Corrected tests in one run
- Compensation of drift effects for long-term measurements
- Corrected RCS (Rate-Controlled Sintering, Super-Res®)

Measurement System

The high-resolution displacement transducer (LVDT, 0.125 nm/1.25 nm/digit) is employed in an Invar measuring system. The maximum measuring range is 5000 μm. The extremely low drift exhibited by the system guarantees measurements with very high repeatability, accuracy and long-term stability for application temperatures up to 2000°C.

Furnaces

Five easily exchangeable furnaces cover the temperature range from -180°C to 2000°C. The forced-air, liquid-nitrogen and water-cooled furnaces offer fast heating and cooling times along with a homogeneous temperature profile over the sample.
Atmosphere – High Vacuum

The vacuum-tight (<10^-4 mbar (10^-2 Pa)) design of the DIL 402 CD allows for careful control of the atmosphere and pure gas conditions. Measurements in inert, oxidizing or reducing atmospheres can be performed under static or dynamic conditions. The gas flow can be controlled by a simple gas control supplement or a mass flow controller system (MFC). In order to provide pure inert conditions for oxygen-sensitive materials, various evacuation systems are optionally available.

Thermostatic Control

To prevent any thermal influence from the furnace or from changes in room temperature, the LVDT system is kept at a constant temperature using a precise thermostat. This guarantees reproducible measurements even in the most sensitive measuring range.
Motorized Pushrod and Automatic Zeroing

The motorized movement of the pushrods and the easy-to-move furnace make it quite simple to insert samples. The pushrods are brought into contact with the samples via a software command which also automatically brings the LVDT to a zero/central position.

Adjustable Contact Pressure

In the DIL 402 CD, the contact force of the pushrod can be adjusted between 15 cN and 45 cN. An almost frictionless ball-bearing design supporting the pushrod guarantees smooth detection of changes in sample dimension.

Evolved Gas Analysis

The vacuum-tight design of the DIL 402 CD is ideally suited for connection to a mass spectrometer via a capillary coupling or to an FT-IR via a transfer line. Outgassing of additives, organic binders and decomposition products can thus be studied.

Tube-Type Sample Carrier

Tube-type sample carriers are available in:

- Fused silica (max. 1100°C)
- Alumina (max. 1680°C)
- Graphite (max. 2000°C)

In addition, a special sample carrier for measurements on thin metal, ceramic and glass foils can be used with the DIL 402 CD.

Sample Geometries

Various sample geometries such as thin rods or plates can be easily measured in the horizontal arrangement. The system allows for measurements on samples with a diameter of max. 6 mm which are between 0 and 25 mm long. In the case of sample decomposition, the design of the transducer system protects it from contamination.
## Technical Key Data for the DIL 402 CD

<table>
<thead>
<tr>
<th><strong>Temperature range</strong></th>
<th>-180°C to 2000°C</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Furnaces</strong></td>
<td>Five user-interchangeable furnaces:</td>
</tr>
<tr>
<td></td>
<td>- Metal: -180°C to 500°C (two versions available; closed or open to one side)</td>
</tr>
<tr>
<td></td>
<td>- Stainless steel: -150°C to 1000°C (with extended heating zone)</td>
</tr>
<tr>
<td></td>
<td>- SiC: RT to 1600°C</td>
</tr>
<tr>
<td></td>
<td>- Rh: RT to 1600°C (for use in glove box, Ar atmosphere)</td>
</tr>
<tr>
<td></td>
<td>- Graphite: RT to 2000°C</td>
</tr>
<tr>
<td><strong>Interchangeable protective tubes</strong></td>
<td>Depending on furnace, available as open or closed tubes; made of:</td>
</tr>
<tr>
<td></td>
<td>- Fused silica</td>
</tr>
<tr>
<td></td>
<td>- Alumina</td>
</tr>
<tr>
<td></td>
<td>- Glassy carbon</td>
</tr>
<tr>
<td><strong>Heating rate</strong></td>
<td>0.01 to 50 K/min</td>
</tr>
<tr>
<td><strong>Temperature precision</strong></td>
<td>0.1 K</td>
</tr>
<tr>
<td><strong>Cooling systems</strong></td>
<td>Liquid nitrogen (low-temperature furnaces)</td>
</tr>
<tr>
<td></td>
<td>Forced-air integrated in SiC furnace and tube furnace</td>
</tr>
<tr>
<td></td>
<td>Water cooling (graphite furnace)</td>
</tr>
<tr>
<td><strong>Atmosphere</strong></td>
<td>Inert</td>
</tr>
<tr>
<td></td>
<td>Oxidizing (graphite furnace up to 1680°C with special protective tube)</td>
</tr>
<tr>
<td></td>
<td>Reducing</td>
</tr>
<tr>
<td></td>
<td>Vacuum (&lt;10⁻⁴ mbar (10⁻² Pa))</td>
</tr>
<tr>
<td></td>
<td>Static and dynamic operation</td>
</tr>
<tr>
<td><strong>Gas flow control options</strong></td>
<td>Gas flow control supplement for 1 gas path</td>
</tr>
<tr>
<td></td>
<td>MFC gas control box for 3 gases</td>
</tr>
<tr>
<td><strong>c-DTA®</strong></td>
<td>For temperature calibration and detection of endo- and exothermal effects</td>
</tr>
<tr>
<td><strong>Measuring range</strong></td>
<td>500/5000 μm</td>
</tr>
<tr>
<td><strong>Load at the sample</strong></td>
<td>15 cN to 45 cN, adjustable</td>
</tr>
<tr>
<td><strong>Δl resolution</strong></td>
<td>0.125 nm/digit, 1.25 nm/digit</td>
</tr>
<tr>
<td><strong>Sample diameter</strong></td>
<td>Up to 6 mm</td>
</tr>
<tr>
<td><strong>Sample length</strong></td>
<td>Up to 25 mm</td>
</tr>
<tr>
<td><strong>Interchangeable tube-type sample holders</strong></td>
<td>Fused silica</td>
</tr>
<tr>
<td></td>
<td>Alumina</td>
</tr>
<tr>
<td></td>
<td>Graphite</td>
</tr>
<tr>
<td><strong>Special sample holder system</strong></td>
<td>Slotted rod for measurements on thin metal, ceramic and glass foils</td>
</tr>
</tbody>
</table>

Sample holder for measurements on thin foils
DIL 402 E – Precision at the Highest of Temperatures

Dilatometer DIL 402 E

For dilatometer measurements in the highest temperature range, the versatile, modular DIL 402 E is the instrument of choice. The design of this instrument is based upon the well-proven dilatometer series, but it can be equipped to achieve two additional upper temperature limits.

Furnaces

Two user-exchangeable furnaces are available, allowing for a broad temperature range and high flexibility. Measurements can be carried out either conventionally with thermocouple control (up to 2000°C), or using the optical pyrometer (up to 2400°C or even 2800°C). The furnace can be slid back and forth on guide rails to allow easy access for uncomplicated insertion or change of the sample.

Design

Depending on the furnace type, various cooling facilities (e.g., forced air or water) are available which offer fast heating and cooling rates along with a homogeneous temperature profile at the sample. The water-cooled graphite tube furnace allows for heating rates of up to 50 K/min and short cooling periods to room temperature.

Safety System

A comprehensive safety system continuously checks the flows of cooling water and protective gas during the measurement. The power supply to the furnace would immediately be cut off if trouble should arise.

Thermostatic Control

To prevent any thermal influence from the furnace or from changes in room temperature, the LVDT system is kept at a constant temperature using a precise thermostat. This guarantees reproducible measurements even in the most sensitive measuring range.
Atmosphere – High Vacuum

The DIL 402 E can be operated under inert, reducing, static or dynamic atmospheres. Various vacuum pumps are available, including turbo molecular pumps for creating the purest of atmospheres. This vacuum-tight dilatometer allows for careful control of the atmosphere and pure gas conditions by using a mass flow controller.

Pushrod and LVDT Systems

The mechanics of the pushrod and transducer (LVDT) are not influenced by the furnace and sample holder. They remain at a constant temperature due to the thermostatic control. The pushrod can apply a load as low as 15 cN to the sample.

Data Acquisition

The transducer’s measuring signal is evaluated in the TA system controller. The rapid analog-to-digital conversion is carried out at 4 million digits and reaches a high resolution of 0.125/1.25 nm/digit.
Temperature Measurement

The instrument version covering a temperature range from 25°C to 2000°C uses a W3%Re-W25%Re thermocouple to control the furnace and to measure the sample temperature. A special thermocouple with a molybdenum protective sheath can be supplied upon request. A special supplement allows temperatures up to 2400°C to be measured using an optical pyrometer (DIL 402 E/7). In addition, a second version (DIL 402 E/8 Pyro) allows for measurements up to 2800°C also with a pyrometer in helium atmosphere. In the temperature range between room temperature and 650°C (i.e., below the working temperature of the pyrometer), the furnace is controlled by an electronic supplement with a ramp generator, signal amplifier and converter. The furnace is heated to 650°C at a constant heating rate of 50 K/min. The change in length is recorded beginning at room temperature, and the correlation to the sample temperature starts at 650°C using the pyrometer.

Optical Pyrometer

The non-contact optical pyrometer measures the temperature at the end of the sample holder by means of a lens system placed in the furnace axis. In this way, the material that is measured always exhibits very similar radiation behavior to that of a black body. The advantage to using this as the temperature measurement point is that variations in the emissivity of different sample materials and surfaces will not influence the measurement. The infrared radiation from the measurement point is collected by the optics and then transmitted through a glass fiber to the IR sensor of the pyrometer electronics. The sensor signals are linearized and digitally displayed in degrees centigrade. Simultaneously, they are sent to the TA system controller to be processed for temperature control and later evaluation.
### Technical Key Data for the DIL 402 E

<table>
<thead>
<tr>
<th>Feature</th>
<th>Details</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Temperature range</strong></td>
<td>Room temperature to 2800°C</td>
</tr>
</tbody>
</table>
| **User-interchangeable furnaces**| - RT to 2000°C (thermocouple operation) to 650°C to 2400°C (pyrometer operation)  
  - RT to 2000°C (thermocouple operation) to 650°C to 2800°C (pyrometer operation) |
| **Interchangeable protective tubes for the 2400°C furnace** | - Al2O3 for tests under oxidative atmospheres up to 1680°C  
  - Glassy carbon |
| **Heating rate**                  | 0.01 to 50 K/min                                                        |
| **Cooling systems**               | Water cooling or cooling thermostat                                      |
| **Atmosphere**                    | - Inert (He > 2000°C)  
  - Oxidizing (graphite furnace up to 1680°C with special Al2O3 protective tube)  
  - Reducing  
  - Static and dynamic operation |
| **Vacuum**                        | <10⁻⁴ mbar (10⁻² Pa)                                                   |
| **Gas flow control options**      | - Gas flow control supplement for 1 gas path  
  - MFC gas control box for 3 gases |
| **c-DTA®**                        | For temperature calibration and detection of endo- and exothermal effects |
| **Measuring range**               | 500/5000 μm max                                                        |
| **Load at the sample**            | 15 cN to 45 cN                                                         |
| **Δl resolution**                 | 0.125 nm/digit, 1.25 nm/digit                                           |
| **Sample diameter**               | 12 mm in thermocouple operation  
  6 mm in pyrometer operation |
| **Sample length**                 | Up to 25 mm in thermocouple or pyrometer operation up to 2800°C         |
| **Exchangeable sample holders and push rods** | - For 2800°C furnace: Graphite  
  - For 2400°C furnace: Graphite, glassy carbon, alumina (<1680°C) |
| **Special sample holder systems** | Sample containers for pastes, liquids and molten metals made of  
  fused silica, alumina and sapphire |
| **Accessories**                   | Sample supports and protective sleeves                                   |

* The 2800°C furnace is operated without protective tube
Dilatometry Accessories

**Sample Containers, Supports and Protective Sleeves**

Special sample containers allow measurements on pastes, powders, molten metals and other material configurations. They are made of fused silica, sapphire, alumina or graphite. Supports come in fused silica, Al$_2$O$_3$ and graphite. Protective sleeves for the sample are available in boron nitride (BN), aluminum nitride (AlN) and molybdenum.

**Atmospheric Control**

The vacuum-tight dilatometer systems (DIL 402 C, CD, E) allow for careful control of the atmosphere and pure gas conditions by using a mass flow controller (MFC). Various evacuation systems are available to support defined, pure atmospheres.
Sample Preparation

Grinding machines for the preparation of plan-parallel samples and vernier calipers for the online input of sample length can be ordered with the instrument.

Calibration

For calibration of the expansion, manufacturer-certified standards are available. These come in different lengths and materials (fused silica, alumina, sapphire, steel, platinum, tungsten, and POCO graphite). All calibration routines are supported by the standard dilatometer software.

Oxygen Trap System (OTS®) for the DIL 402 C

The presence of residual oxygen can be critical in certain applications (e.g., metals, metal alloys) because possible oxidation of the sample would lead to undesired results and false interpretations. The OTS® system allows for effective reduction of the oxygen partial pressure in the vicinity of the sample. A ceramic substrate bearing a getter ring is mounted on the sample carrier or in the sample carrier tube. The residual oxygen content remaining after evacuation is thereby almost entirely eliminated (< 1ppm).

Additional information

See brochure: Accessories for Thermomechanical Analysis
**Proteus® Software for Dilatometry**

All dilatometers run under Proteus® software on a Windows® operating system. User-friendly menus combined with automated routines make Proteus® very easy to use while still providing sophisticated analysis. The Proteus® software is licensed with the instrument and can of course be installed on other computer systems.

**Key Features of the General Software**

- Fully compatible with other Microsoft® Windows® programs
- Multi-tasking: simultaneous measurement and evaluation
- Multi-moduling: operation of different instruments with one computer
- Context-sensitive help system
- Labeling: input and free placement of text elements
- Storage of the analysis results and status for later restoration and continuation with analysis (original file remains)
- Storage of c-DTA® curves which can be directly accessed, as ‘true’ measurement files
- Results by e-mail
- Freely configurable ASCII import, import of measurement files for coupled methods (QMS and FT-IR data); data is linked to the DIL temperature curve
- Export of graphics with evaluation results to clipboard or to common formats such as EMF, PNG, BMP, JPG, TIF or PDF
- ASCII file export of the raw data and/or the corrected measurement data for data processing with more extensive analysis programs
- Data can be exported into Excel®-compatible CSV-format
- Multiple-window technique for clear presentation and evaluation of measurement curves or graphical excerpts in multiple windows
- Formatting of the results, measurement values, and axis labels optionally in technical format or as scientific notation
- Application languages: English, German, French, Russian, Chinese
Key Features of the Measurement Software

- Repetitive measurements with minimum parameter input
- Temperature program: up to 256 programmable temperature segments
- Programming while the measurement is running
- Monitoring of all MFC gas flows (protective and purge gas)
- Loop programming: insertion, deletion, and annexation of temperature segments, even in already existing temperature programs
- Fast definition for furnace heating

RCS test to achieve a constant shrinkage rate
Key Features of the Evaluation Software

- Presentation/evaluation of the absolute (dL in μm, corrected or raw data) or relative length change curve (dL/Lo or dL/Lo in %)

- Calculation of 1st and 2nd derivative

- Correction of the measured length change either via sample holder expansion or using a calibration run

- Parallel curve offset (offset correction)

- Corrections as per ASTM (for samples and calibration standards of identical length) or as per DIN (for samples and calibration standards of not necessarily identical length)

- Correction possibility which allows the reference temperature Tref (Lo) to be shifted to values other than 20°C (DIN)

- Offset correction for start value and/or initial sample length can be carried out with offsets manually defined, automatically read from raw data or by graphical extrapolation

- Calculation/determination of individual values and graphical presentation of the technical and/or physical expansion coefficient

- Possibility of extrapolating the coefficient of technical expansion (CTE) to reference temperatures between 20°C and 50°C

- Comparative analysis of up to 64 curves or segments from the same or different measurements

- Value determination on a single curve or a curve family

- Semi-automatic routines for determination of characteristic temperatures of the length change curve, its derivative and/or the curve of the expansion coefficient, peaks; simultaneously on multiple curves

- Smoothing of the measuring curves with adjustable filter factors

- Glass transition evaluation

- Evaluation of sintering steps

- Search for temperature for a given percentage length change

- Automatic softening point detection (programmable, measurement stop or jump into the next segment of the temperature program)

- Connection of segments by spline interpolation

- Snapshot: Online evaluation of the running measurement

- Comparative evaluation and presentation of various signals and methods (TGA, DSC, DTA, STA, DIL/TMA, DMA, mass spectrometry and FT-IR) in collective graphics or evaluation windows

- Macro-recorder for creating analysis macros (“learning by doing”) and for the automatic evaluation of measurement series. Results can additionally be annotated with threshold values for quality control

- Expanded evaluation function for imported mass spectrometer data from coupling with QMS 403 C Aëolos®. The MS data is linked to the dL/Lo data time-wise and temperature-wise, incl. 3D presentation together with temperature and dL/Lo-signal
Software Specialties

- **Patented c-DTA®**
  The optionally available software add-on c-DTA® ("calculated DTA") allows for simultaneous analysis of length changes, endothermal/exothermal effects, and temperature calibration.

- **Rate-Controlled Sintering (RCS)**
  The RCS software add-on is a rate-controlled temperature guidance for optimization of the sintering process. Three different modes are included: start/stop, stepwise isothermal, and dynamic heating rate.

- **Peak Separation**
  For accurate separation and evaluation of overlapping effects, this optional software program can also be used for further thermoanalytical and gas analysis measurements such as DSC, TGA, QMS, FT-IR, etc.

- **Density Determination**
  This optional program add-on supports determination of the density of solid samples as a function of temperature beyond the melting temperature. The software includes volume expansion and can also be used for liquids and pastes.
Performance and Low-Temperature Applications

Unmatched Reproducibility

Here, a sapphire sample was measured twice in the direction of the c-axis during heating and cooling between room temperature and 1550°C. The heating/cooling rates were 5 K/min. The atmosphere was helium. The comparison clearly demonstrates the outstanding reproducibility of the DIL 402 C. As can be seen, the heating and cooling data are almost identical. The difference between the four test results is typically less than 0.3%.

Outstanding Accuracy

In this example, the linear thermal expansion behaviors of pure aluminum, copper and electrolytic iron are compared with literature values. Clearly there is excellent agreement. The differences are generally less than 1%. In addition, Pt/10%Rh was compared with the values for platinum and rhodium. The test results are in the expected range between the pure metals.

Excellent CTE Repeatability – Glass Ceramic

ZERODUR® is a glass ceramic produced by SCHOTT, Germany. It is designed for zero thermal expansion around room temperature. This material is often used for high-performance terrestrial telescopes. The figure shows the linear thermal expansion between -150°C and 100°C. The sample was measured twice at a heating rate of 3 K/min in a helium atmosphere. The measured CTEs between 0°C and 50°C are in excellent agreement with the literature values (SCHOTT brochure) for this material.
Fiber-Reinforced Polymer

A two-dimensional fiber-reinforced polymer was measured in and perpendicular to the fiber orientation. Both tests were carried out between -100°C and 100°C. In the fiber direction, the CTE is strongly reduced by the influence of the fibers. Perpendicular to the fibers, their influence is small; the CTE is in the typical range for the polymer matrix. Additionally, the glass transition of the polymer is clearly visible in the perpendicular direction. This example shows that fiber reinforcement has a significant effect on the thermal expansion behavior.

Roof Tiles

A fired roof tile was exposed to water for 24 hours and then tested with the DIL 402 C between -20°C and 25°C. Upon cooling, the water in the pores of the ceramic body freezes at -7°C. (The low freezing temperature is due to supercooling of the water.) The freezing causes an increase in the sample length of nearly 0.08%. Upon heating, the ice begins melting at -2°C, resulting in a shrinkage of the sample. The irreversible length change of the sample after cyclic cooling and heating may be partially due to cracks. The use of this roof tile type in certain climate zones should be given careful consideration prior to installation. Freezing effects will reduce its lifetime.

The DIL 402 C is also suitable for tests on polymer materials. The possibility of running controlled heating and cooling cycles in the DIL 402 C results in a better understanding of the material’s behavior.
High-Temperature Applications

Determination of the Softening Point – Quality Control

Coefficients of thermal expansion (CTE), glass transition temperatures and softening points are crucial parameters for characterization of glass materials. This plot shows three tests on the same type of glass but from different batches. It can clearly be seen that the coefficients of the thermal expansion are in good agreement within the instrument’s uncertainty boundaries. The glass transition temperatures and the softening point of sample no. 3 (blue curve) show lower values, indicating a slightly different composition. The thermal expansion tests were, of course, switched off automatically by the softening point detection in order to protect the system from contamination.

Brick Clay

Brick and tile products contribute significantly to the development and quality of construction projects all over the world. This figure of a brick clay measurement exhibits a step in the thermal expansion curve (red) at 576°C which can clearly be seen in the physical α-curve as a peak at 587°C. This change in thermal expansion is due to the overlapping dehydration of clays and phase transition of quartz. Above 800°C, sintering starts. During the sintering process, a melting effect can be observed which could be due to an inorganic component of the raw material.
High Sample Throughput –
Fired Tiles with Different Expansion Behavior

Two colored tiles were simultaneously heated at 3 K/min to 1100°C using the DIL 402 CD. The curves for the relative expansion of these fired tiles show significant differences in expansivity and quartz content (>567°C).

The powerful NETZSCH Proteus® measuring and evaluation software allows a graphic display of the expansion difference in a separate curve.
High-Temperature Applications

Comparison Between an Already Sintered Zirconia and Its Green Body

Zirconia is used as a refractory material in insulation, abrasives, enamels and ceramic glazes, but it is also used in the manufacture of subframes for the construction of dental restorations. This plot compares an already sintered material with a zirconia green body. While the sintered material shows a nearly linear thermal expansion, the green body still exhibits the binder burnout at 389°C and sintering which starts at 970°C. The highest sintering rate is achieved at 1403°C (dL/dt curve).

With the DIL 402 CD, comparative measurements are easy. The comparison between the green body and sintered zirconia is demonstrated here.
Silicon Nitride

Because of its excellent thermal and mechanical properties, silicon nitride is used increasingly often in high-tech applications (e.g., valves in automobile engines). Of course, the properties of the final parts are heavily influenced by the production/sintering process. Depicted in this plot are the thermal expansion and rate of expansion of a silicon nitride green body. The sintering step starting at 1201°C is due to the influence of sintering additives. The major shrinkage step occurred at 1424°C (extrapolated onset). The effect above 1760°C is most probably due to the evaporation of additives.

Natural Carbon

The reduction of impurities in natural carbon can be seen during heating under a helium atmosphere. The sintering behavior changes during reduction, which can be observed in the effects between 1150°C and 1600°C. After these effects, the thermal expansion increases almost linearly up to 2600°C. Reliable measurements can be performed at the highest temperatures with the DIL 402 E up to 2800°C. The temperature is monitored with a pyrometer.

DIL 402 C allows measurements under well-defined atmospheres up to 2000°C. This allows observation of the complete sintering behavior of many ceramic materials.

The purest of gas atmospheres and highest of temperatures can be achieved with the DIL 402 E.
c-\textit{DTA®} and Other Specialties – Deep Insight into Material Behavior

\textbf{c-\textit{DTA®} Feature}

The \textit{c-\textit{DTA®}} calculation is a mathematical routine based on the temperature measurement at the sample. Transitions associated with exo- or endothermal effects, of course, slightly influence the temperature change during dynamic heating or cooling. By comparing the measured temperature change of the sample with a theoretical one, these exo- or endothermal effects can be detected. Therefore, more detailed insight into the material behavior during expansion, shrinkage and sintering can be obtained. If a calibration run on a standard sample is carried out prior to the sample run, this will also be taken into consideration in the calculation process. In addition, \textit{c-\textit{DTA®}} is used for temperature calibration of the dilatometer systems.

\textbf{Production of a Cordierite Ceramic}

During the production of cordierite (an alumina-silica ceramic carrier for catalysts), various raw materials are ground and mixed to form a green body. This plot shows the simulation of the firing process of a cordierite raw material. Under oxidizing atmospheres, the organic additives are burnt out. Using the \textit{c-\textit{DTA®}} software, the burnout of the additives can be observed as an exothermic effect between 190°C and 525°C (blue curve). At high temperatures (> 890°C), sintering starts and the cordierite phase is formed.

Simulation of the production process of cordierite by using the DIL 402 PC in combination with \textit{c-\textit{DTA®}} (sample length 24.83 mm, heating rate 5 K/min)
Volumetric Expansion and Bulk Density of Powders, Pastes and Liquid Metals

Dilatometers are generally used for measurement of solids. However, by using the liquid metal containers (made of fused silica, graphite, alumina or sapphire) and a special software extension, the volumetric expansion and bulk density of powders, pastes and liquid metals can now be accurately determined. Knowledge of the thermophysical properties such as volumetric expansion and bulk density of metals during melting is of paramount importance for the simulation of casting processes using finite element models.

Superalloy – Inconel 718

The volumetric expansion and bulk density of a nickel-based superalloy (Inconel 718) were measured in the solid and liquid regions as well as in the "mushy" zone. A sapphire container was employed for this test. The influence of the container on the result was corrected by the NETZSCH Density Determination software. The onset of melting of the sample was detected at 1292°C. The melting process was finished at 1346°C. The volume change during melting was 3.1%. Since the room temperature bulk density and thermal expansion were known, it was possible to calculate density as a function of temperature.

The NETZSCH Density Determination software allows calculation of the density as a function of temperature based on the thermal expansion.
Cast Iron – Volume and Density Change

Cast iron is a ferrous alloy which has been heated until it liquefies, and is then poured into a mold to solidify.

In this plot, simulation of the expansion behavior during the casting process of a cast iron sample was realized by using a sample container made of Al₂O₃. Nearly a constant expansion behavior \( \frac{dL}{L_0} \) was observed until melting of the sample at 1334°C (extrapolated onset). During melting, a big leap is monitored for the expansion \( \frac{dL}{L_0} \) and the technical expansion coefficient \( \alpha \). The sample is completely molten at 1370°C (extrapolated endset).

The volumetric expansion and temperature-dependent density change were calculated using the software module Density Determination. During melting, the sample experienced an expansion step of more than 4% within a temperature difference of 40°C.

Measurements on a cast iron sample from the solid state into the liquid phase

Density determination of a cast iron sample
Dilatometry under Humidity

For the DIL 402 C, a humidity generator is available for measurements under a relative humidity of between 5% and 90% in the temperature range from 30°C to 80°C.

Measurements in a humid atmosphere are carried out with a rod-type sample holder for optimum access of the humid atmosphere to the sample surface.

In place of the standard rod-type sample holder, a special rod-type sample holder with a humidity sensor is available.

Influence of Humidity on a Polymer

Here, a polymer sample (length 11.8 mm) was measured with the DIL 402 C connected to the humidity generator. The temperature was kept constant at 55°C. The atmosphere was preset to nitrogen with 70% humidity. After switching on the humidity generator, the sample immediately expands in two steps. After 200 min, the humidity generator is switched off and the sample begins drying. The drying process can be observed in the subsequent shrinkage of the sample. However, even after 15 h of the drying process, the sample does not shrink back to its initial length.

DIL 402 C measurement on a polymer under humid conditions up to 200 min.
The NETZSCH Group is an owner-managed, internationally operating technology company headquartered in Germany.

The three Business Units – Analyzing & Testing, Grinding & Dispersing and Pumps & Systems – provide tailored solutions for highest-level needs. Over 2,500 employees at 130 sales and production centers in 23 countries across the globe guarantee that expert service is never far from our customers.

When it comes to Thermal Analysis, Adiabatic Reaction Calorimetry and the determination of Thermophysical Properties, NETZSCH has it covered. Our 50 years of applications experience, broad state-of-the-art product line and comprehensive service offerings ensure that our solutions will not only meet your every requirement but also exceed your every expectation.