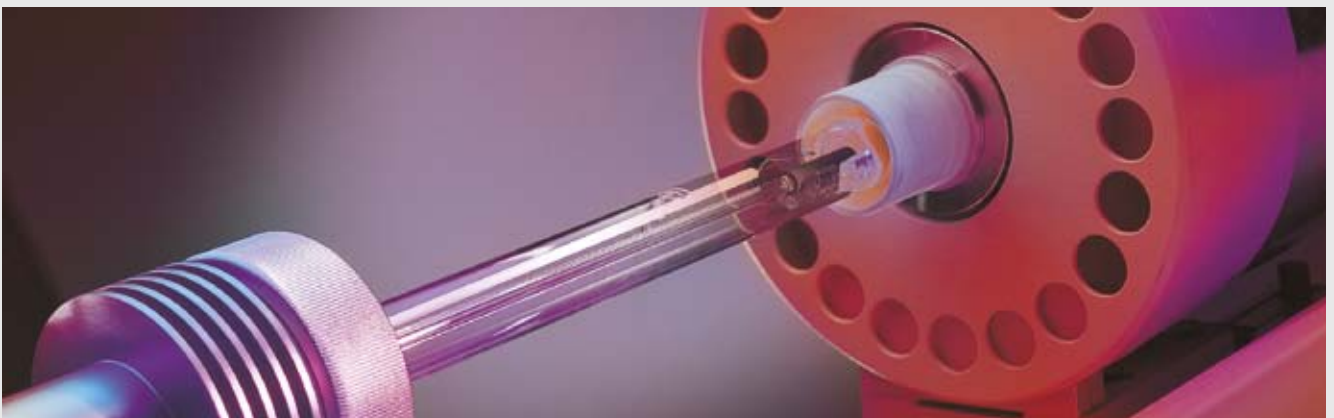
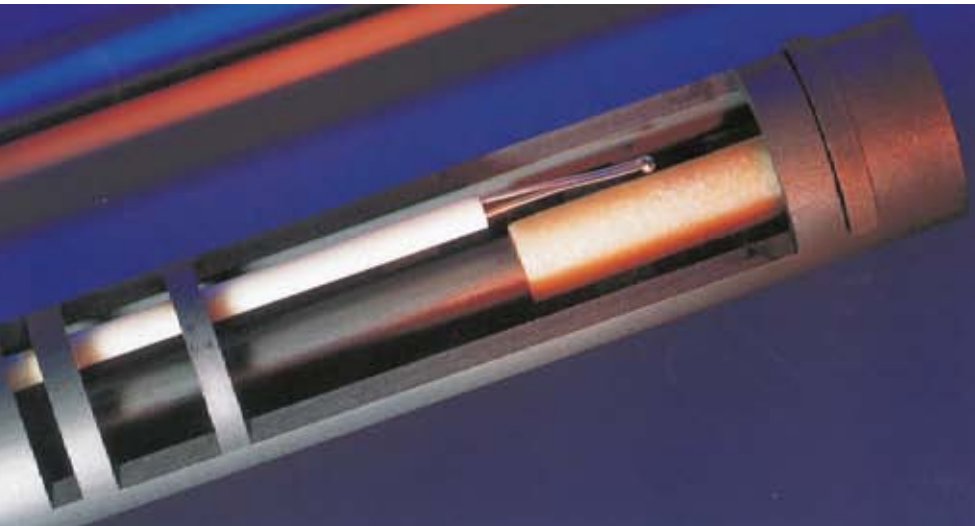


Dilatometry

Method, Instruments, Applications – -180°C ... 2800°C



Dilatometry – Method



Typical arrangement of a sample (beige) in a dilatometer sample carrier (graphite), the thermocouple (with white capillary tube) is located in direct proximity to the sample.

Dilatometry (DIL) is a thermoanalytical technique for the measurement of expansion or shrinkage of a material when subjected to a controlled temperature/time program.

The method is standardized and operates on bases such as DIN EN 821, DIN 51045, ASTM E 831 and ASTM E 228. Besides solid samples, powders, pastes and even liquids can be tested in special containers.

DIL Measurement Information

- Linear thermal expansion
- Determination of the coefficient of thermal expansion (CTE)
- Sintering temperature and sintering step
- Volumetric expansion
- Density change
- Determination of glass transition temperatures
- Softening points
- Phase transitions
- Influence of additives and raw materials
- Optimizing of firing processes
- Kinetic studies
- Rate-controlled sintering (RCS)



DIL 402 C as an example of a pushrod dilatometer

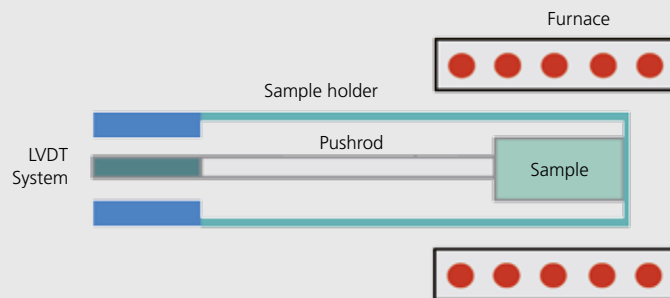
Functional Principle

A pushrod is connected to an inductive displacement transducer (LVDT system) on one side, and on the other, it is in close contact with a sample in order to register any length change in the sample material during heating or cooling.

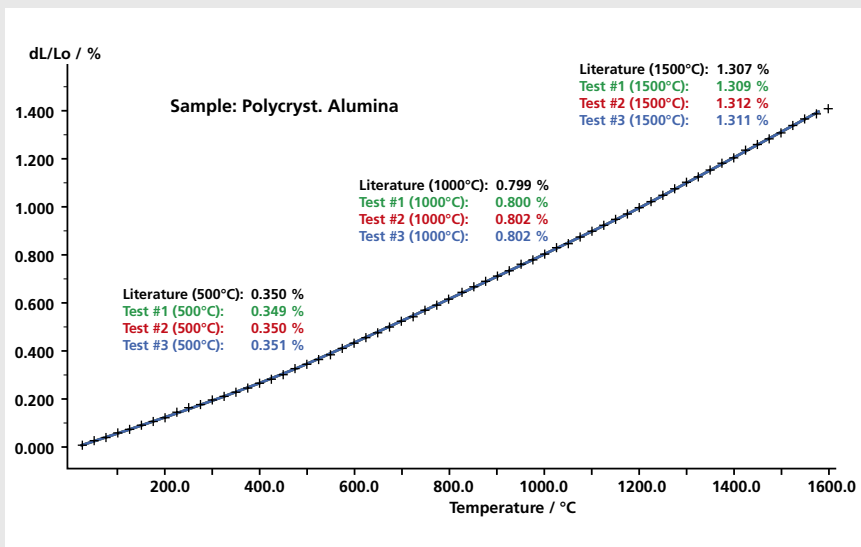
Since the sample holder and the anterior part of the pushrod are exposed to the same temperature program as the sample, they also undergo expansion. The resulting dilatometer signal is therefore the sum of the length changes of the sample, sample holder and pushrod.

To obtain the true sample behavior, it is thus necessary to correct the raw dilatometer data. There are two possible correction methods: to use

tabulated expansion data for the sample holder material or to use a correction curve – the latter of which is often more precise.



Functional diagram of a pushrod dilatometer



Comparison between the expansion data of polycrystalline alumina and literature values

Results and Measuring Accuracy

The thermal expansion in percent is an important value for evaluating the performance of a material.

In the diagram, the length changes for three test runs (lines) on polycrystalline alumina (Al_2O_3) are presented together with the corresponding data from literature (crosses). The differences at 500°C, 1000°C and 1500°C are all lower than 1%, thus demonstrating the outstanding accuracy of the used NETZSCH DIL 402 PC.

DIL 402 C

The Vacuum-Tight Pushrod Dilatometer DIL 402 C – The Perfect Instrument for all Demanding Measurement Tasks

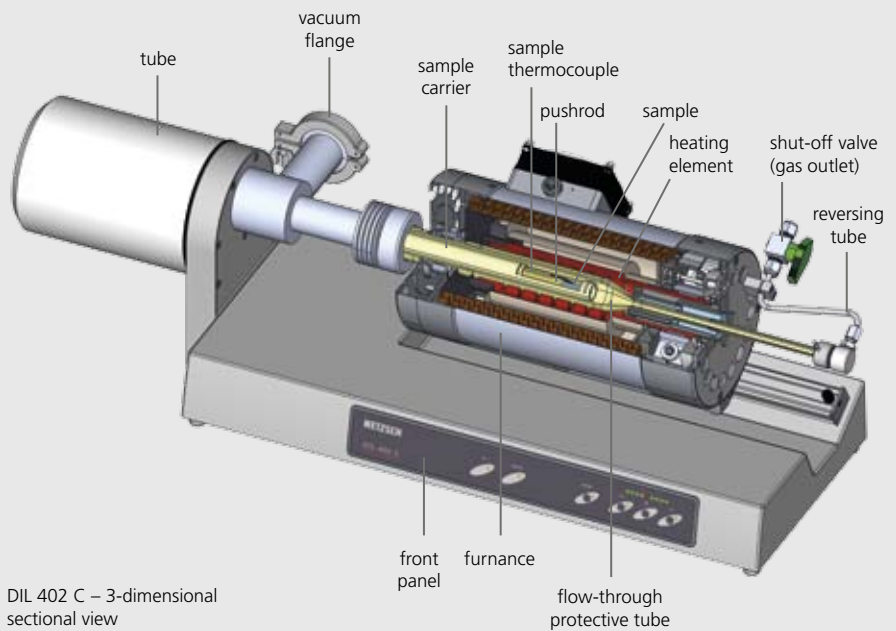
The DIL 402 C is the result of 50 years' worth of experience in building dilatometers at NETZSCH.

This premium instrument has been optimized for application in the traditional areas of ceramics and glass as well as for research in fields such as advanced ceramics or powder metallurgy.

The DIL 402 C has an Invar measurement system with a high-resolution measurement transducer and comprehensive thermostatic control, offering the highest degree of accuracy, reproducibility and long-term stability for application temperatures up to 2000°C.

The instrument's horizontal design with motorized pushrod and easy-to-move furnace makes it simple to insert samples, even when sample geometries are less than ideal. A thermocouple in direct proximity to the sample enables reproducible temperature measurement. This allows for use of the *c-DTA*[®] software to calculate endothermic and exothermic effects in the sample.

Thus, in addition to determining characteristic expansion values, it is also possible to gather additional information about phase transitions, solid-state reactions or chemical reactions such as oxidation.



DIL 402 C – 3-dimensional sectional view

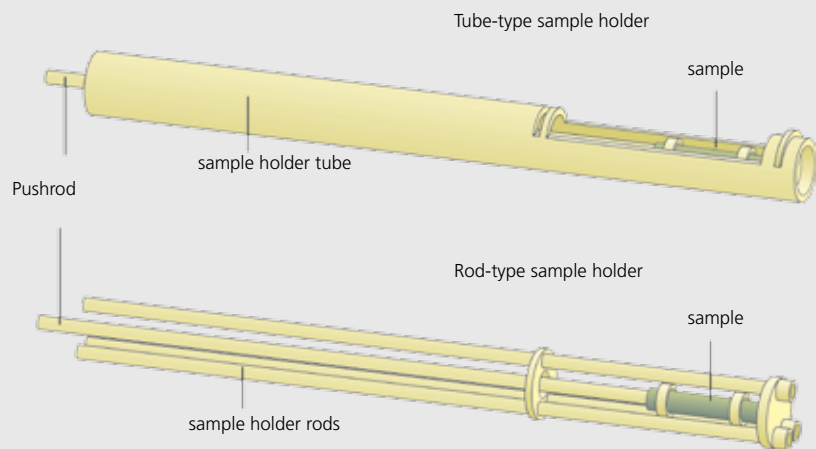
At a glance

The DIL 402 C is characterized by its:

- Wide temperature range (-180°C to 2000°C via exchangeable furnaces)
- Large sample dimensions (length: up to 50 mm, diameter: up to 19 mm)
- Adjustable contact pressure (15 cN to 45 cN)
- Extremely low drift, high resolution
- Vacuum-tight design
- Capability of coupling to gas analysis systems like QMS or FTIR

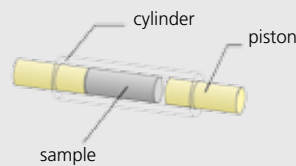
Sample Carriers

The tube-type sample carriers are standard and available in fused silica (max. 1100°C), alumina (max. 1680°C) or graphite (max. 2000°C). Supports can be used for centering the sample. The tube-type sample carriers can optionally be replaced with rod-type sample carriers for ideal heat transfer to the sample. They consist of three support rods, leaving the sample open to the heat source and the gas atmosphere. As a result, the accuracy is increased. The rod-type carriers are available in fused silica and alumina.



Special Sample Container

To allow measurements on pastes, powders, molten metals and other material configurations, special sample containers made of fused silica, alumina, sapphire or graphite are optionally available.



Sample container for pastes, powders and molten metals



DIL 402 C

DIL 402 PC

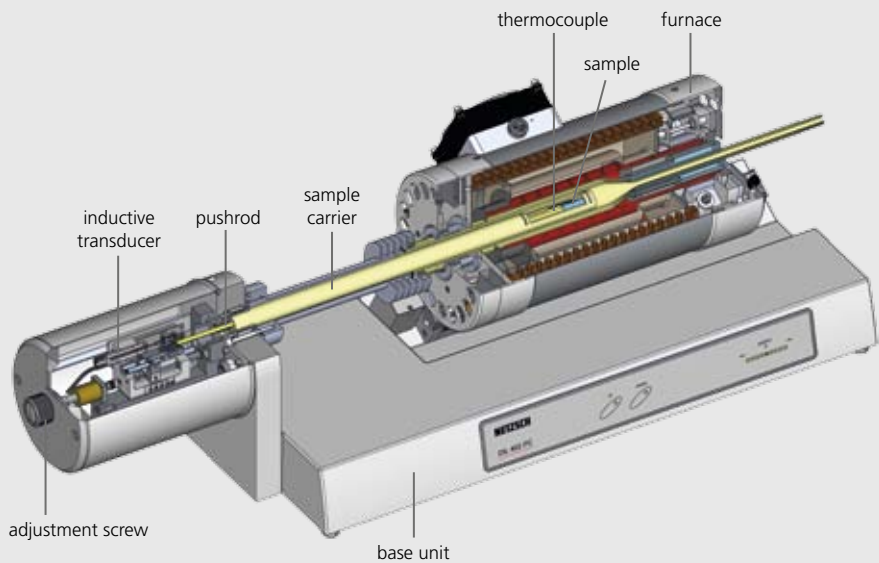
DIL 402 PC – Precise Expansion/Shrinkage Measurement in Routine Operation

The DIL 402 PC is specially tailored to the needs of the ceramics and glass industry. High resolution and stability, a wide measurement range, and a robust and compact construction are only some of the advantages of this cost-effective instrument.

The optimized design of the measurement system with inductive displacement transducer offers a high degree of reproducibility.

Interchangeable furnaces with temperature ranges up to 1200°C and 1600°C serve to allow for diverse applications in expansion tests on solids, green bodies, powders and pastes in the areas of quality and production control.

With automatic softening point detection (available for all NETZSCH dilatometers), even samples which exhibit a tendency to melt (such as glasses or glazes) can be run without any special care. As soon as a defined threshold value is reached, the software either stops the measurement or jumps directly to the next segment of the temperature program.



DIL 402 PC – 3-dimensional sectional view

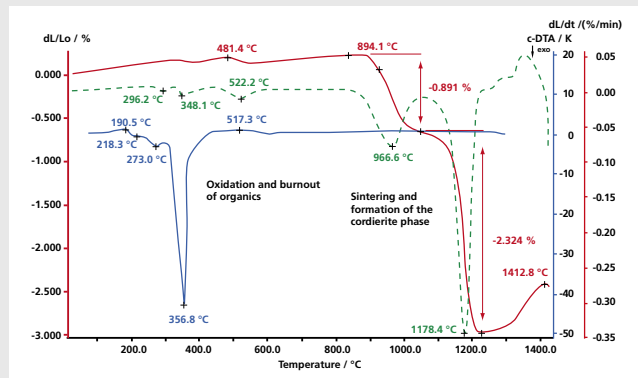
At a glance

The DIL 402 PC is characterized by its:

- High flexibility, two exchangeable furnaces, various accessories
- Large sample dimensions (length: up to 50 mm, diameter: up to 19 mm)
- Ease of operation
- Highly reproducible results

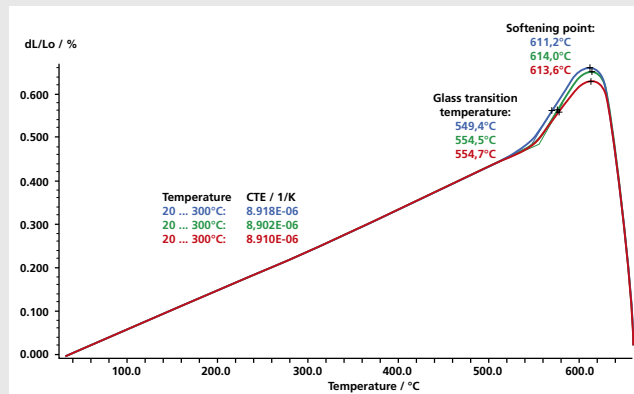
Simulation of the Production Process of Cordierite

During firing of the green body under oxidizing atmospheres, the organic additives are burnt out and the cordierite phase is formed at high temperatures. Using the DIL 402 PC in combination with the *c-DTA*® software (also available for DIL 402 C and DIL 402 CD), the production process can be analyzed in detail.



Glass

Presented in the figure are three tests on same type of glass but from different batches. It can clearly be seen that the coefficients of thermal expansion are in good agreement within the instrument's uncertainty boundaries. The glass transition temperature and the softening point of sample #3 (blue curve) show slightly lower values, indicating a slightly different composition.



DIL 402 PC

DIL 402 E / DIL 402 CD

DIL 402 E – Reliable Thermal Expansion Measurements up to 2800°C

The versatile high-temperature dilatometer DIL 402 E is available in several versions:

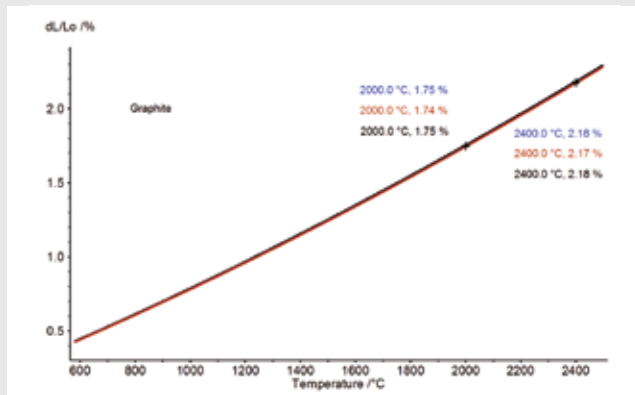
- The model that operates up to 2800°C works with an optical pyrometer which is capable of determining the temperature of the end of the sample holder without establishing contact. This ensures that the emissivity of the sample material itself has no influence on the measurement result.
- The design of the 2400°C model is analogous to that of the 2800°C version. Only the length of the graphite furnace and the heating elements differ.
- In the 2000°C model, the furnace control and the measurement of the sample temperature take place via W/Re thermocouples.

By replacing the graphite or glassy carbon sample holder and protective tube of the graphite furnace with alumina ones, the system can even be operated in an oxidizing atmosphere up to 1680°C.

In all cases, a sophisticated safety system continuously checks the flow of the cooling water and of the protective gas to assure safe operation.



DIL 402 E



Comparison of the expansion behavior of two POCO graphite measurements (blue and red) with the corresponding data from literature (black). The results at 2000°C and 2400°C show excellent agreement with each other.

At a glance

The DIL 402 E is characterized by its:

- Highly flexible modular concept
- High accuracy up to 2800°C
- Simple conversion from one model into another
- Short cooling times back to room temperature
- Vacuum-tight design
- Capability of mass spectrometer coupling

Further information

www.netzsch.com/dil402e

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

Whenever it is important to have a higher sample throughput, a direct comparison of samples (dual version), an online calibration correction of expansion data, or an improved long-term drift stability (differential dilatometer), the DIL 402 CD is the first choice.

The horizontal arrangement of the dilatometer offers specific advantages over the samples with regard to temperature profile, sample handling, and safety. 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.

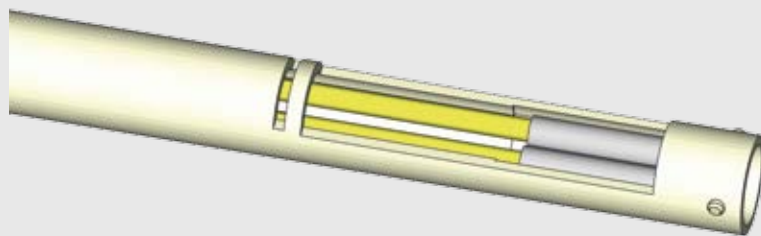
In the event of sample decomposition or melting, the transducer system is effectively protected by its design.

The system allows for measurement of samples up to 25 mm in length and up to 6 mm in diameter, but other geometries such as thin rods or plates can also be accommodated.

Motorized movement of the pushrods and automatic zeroing of both sample positions are standard features of the instrument.



DIL 402 CD



DIL 402 CD sample holder for two samples (sample thermocouple sandwiched between two samples not shown here).

At a glance

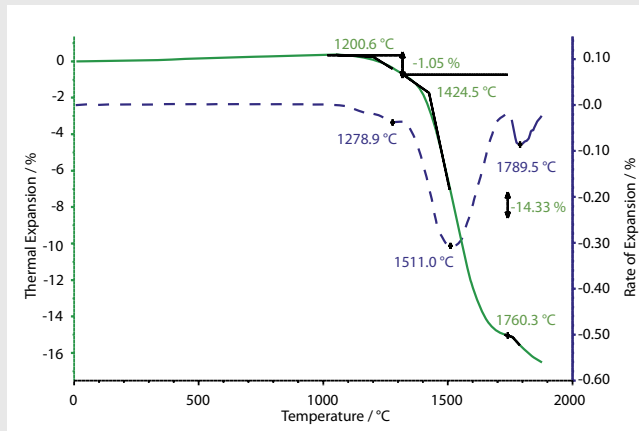
The DIL 402 CD is characterized by its:

- Wide temperature range (-180°C to 2000°C via exchangeable furnaces)
- Two measurement positions
- Vacuum-tight design
- Homogeneous temperature profile
- Capability of evolved gas analysis

Further information

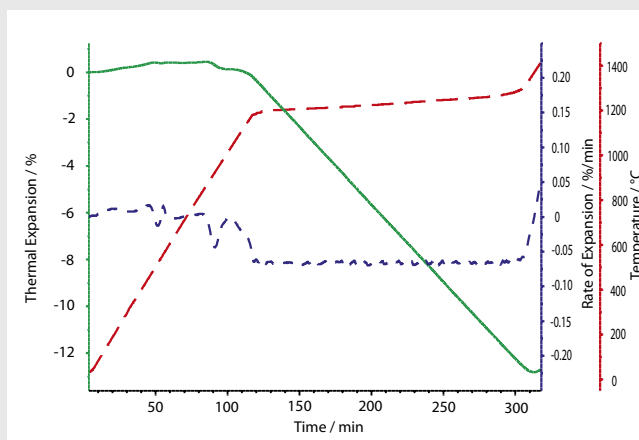
www.netzsch.com/dil402cd

Applications and Technical Specifications



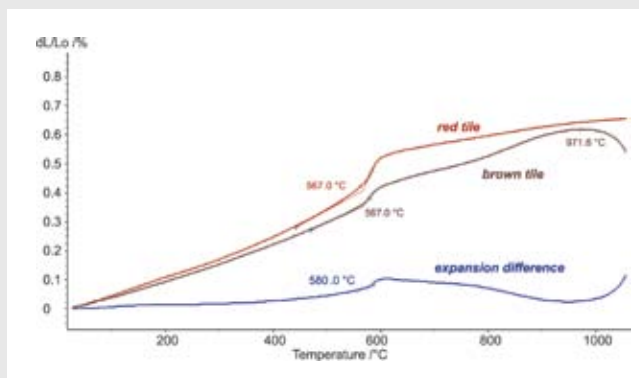
Silicon Nitride

The properties of final ceramic parts are often heavily influenced by the production/sintering process. Depicted in this figure are the thermal expansion and rate of expansion of a silicon nitride green body measured with the DIL 402 C. The sintering step starting at 1201°C can be attributed to the influence of the sintering additives. The main shrinkage step occurs at 1424°C (extrapolated onset temperature). The effect above 1760°C is most probably due to evaporation of additives.



Alumina Green Body

In this example, an alumina green body was tested with the DIL 402 C, employing the NETZSCH Rate-Controlled Sintering (RCS) software in the start/stop mode. The aim of the RCS mode in this case was to achieve a constant shrinkage rate by reducing the heating rate, if necessary. Up to 1150°C, the influence of additives (e.g., organic binder, clays) is visible; the main sintering step occurs between 1150°C and 1350°C.



Tiles with Different Expansion Behavior

Here, 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 (brown and red) show significant differences in expansivity and quartz content (> 567°C). The powerful NETZSCH *Proteus*® measurement and evaluation software allows a graphical display of the expansion difference in a separate curve (blue).

Technical Specifications (subject to change)

	DIL 402 C	DIL 402 PC	DIL 402 CD	DIL 402 E
Temperature range	-180°C to 2000°C	RT to 1600°C	-180°C to 2000°C	RT to 2800°C
Furnaces	-180°C to 500°C -150°C to 1000°C RT to 1000°C RT to 1600°C RT to 2000°C Furnaces interchangeable with DIL 402 CD	RT to 1200°C RT to 1600°C	-180°C to 500°C -150°C to 1000°C RT to 1600°C RT to 2000°C Furnaces interchangeable with DIL 402 C	RT to 1600°C RT to 2400°C RT to 2800°C (data from 700°C upwards) Up to 2000°C with sample thermocouple
Sample holder	Fused silica, alumina, graphite	Fused silica, alumina	Fused silica, alumina, graphite	Graphite, glassy carbon, alumina
Sample dimension	Length: up to 50 mm ø: max. 19 mm Standard ø: max. 12 mm	Length: up to 50 mm ø: max. 19 mm Standard ø: max. 12 mm	Length: up to 25 mm ø: max. 6 mm	Length: up to 25 mm (up to 1680°C up to max. 50 mm length) ø: max. 12 mm (6 mm for pyrometer)
Gas atmosphere	Inert, oxid. (to 1680°C), red., vac.	Oxid. (static/dynamic), purging inert	Inert, oxid. (to 1680°C), red., vac.	Inert (He > 2000°C), oxid. (with alumina protective tube up to 1680°C)

All NETZSCH dilatometers are equipped with *Proteus*[®] software, including softening point detection and – optionally – *c-DTA*[®], Rate-Controlled Sintering (RCS), Density Determination or Thermokinetics.



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.

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