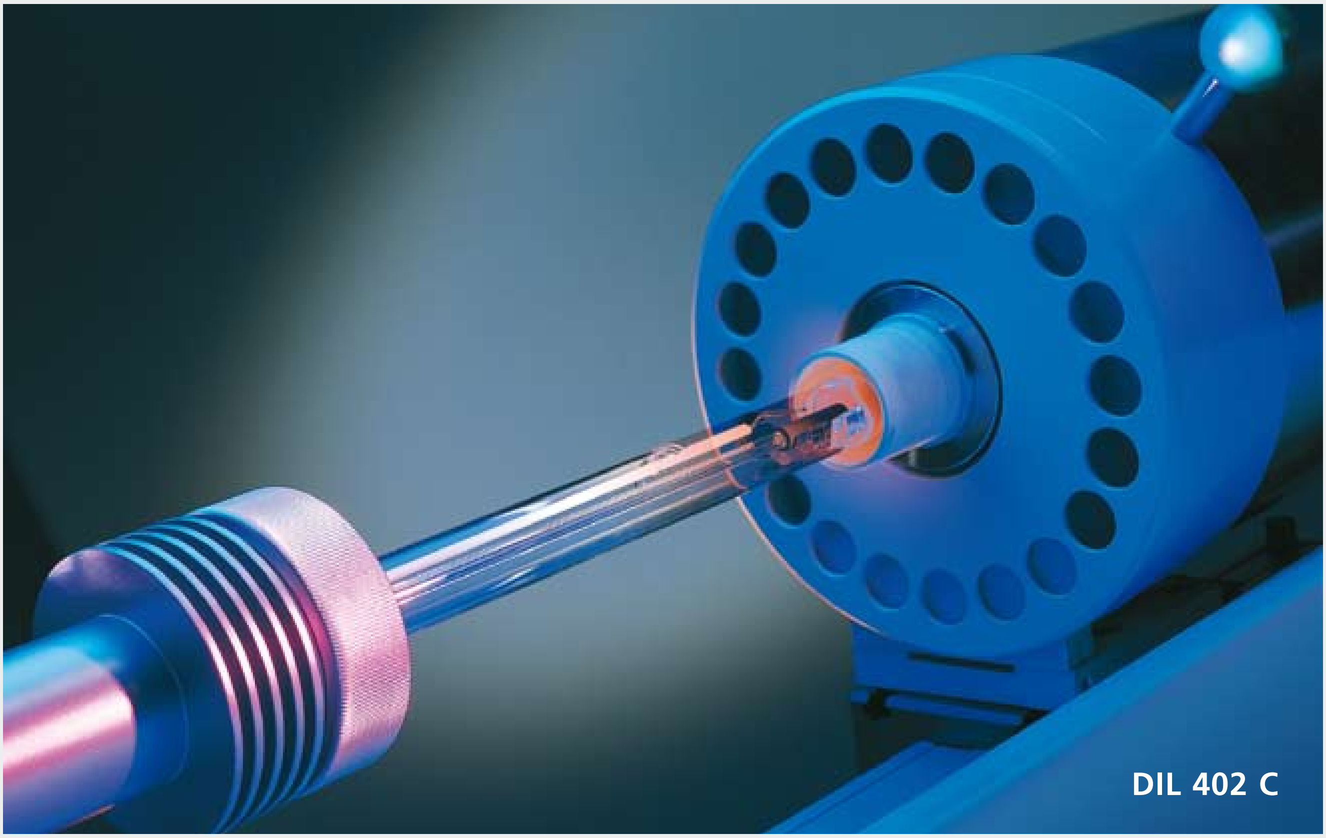
NETZSCH

Analyzing & Testing
Business Unit

Dilatometry
-180 °C to 2000 °C



Leading Thermal Analysis.

Dilatometry - DIL

Dilatometry is a thermo-analytical technique for the measurement of expansion or shrinkage of a material when subjected to a controlled temperature/time program.

Thermal expansion can be accurately measured with the Dilatometer 402 C. The system can also be used to examine phase transitions, solid-state reactions and chemical reactions such as oxidation.

Accurate measurement of dimensional changes is required in the traditional ceramics and glass industries and for carrying out sintering studies on reactive powders used in the fields of advanced ceramics or powder metallurgy.

There are many application areas for dilatometry on solids, powders, pastes and liquids in research and development and production environments:

- Linear thermal expansion
- Determination of the coefficient of thermal expansion (CTE)
- Expansivity
- Sintering temperature and shrinkage steps
- 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)

All these areas have specific requirements and the, NETZSCH Dilatometer 402 C with its excellent balance of features has been optimized to fulfill them all. These include flexibility of temperature range, sample holder and gas atmosphere combined with high accuracy and easy handling. This hardware and software excellence is a result of more than 40 years´ experience in building dilatometers. The DIL 402 C works according to the most common standards, such as DIN 51 045, and ASTM E 831 and ASTM E 228.

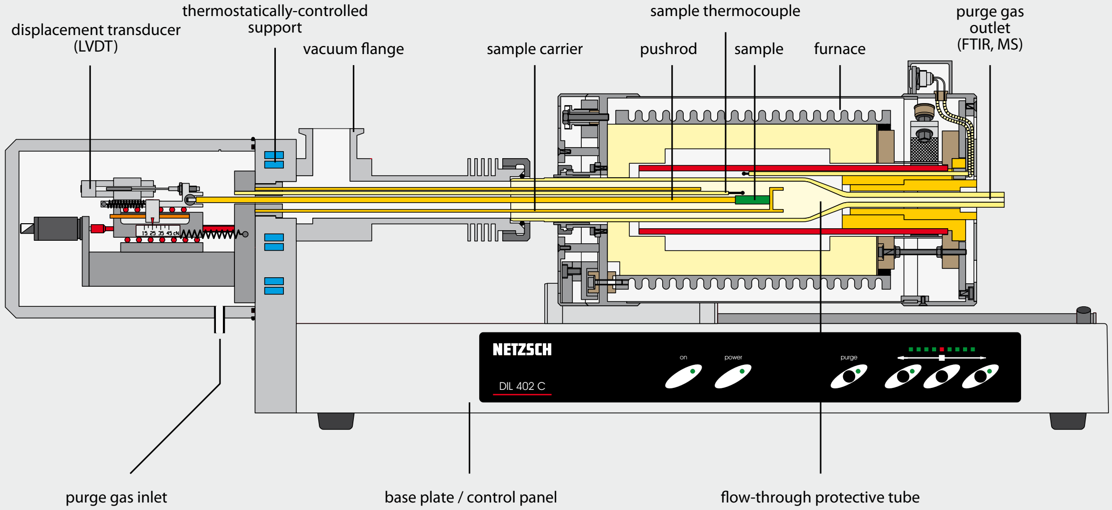
Thermophysical Properties (TPP)

The DIL 402 C is also part of the NETZSCH TPP product line. This also includes the LFA 427 (thermal diffusivity) and the DSC 404 C *Pegasus*® (specific heat). Please refer to the relevant brochures for details.

Dilatometer DIL 402 C (1600 °C model)

DIL 402 C - Hardware

The DIL 402 C carries forth the successful NETZSCH series of horizontal pushrod dilatometers. The state-of-the-art design is compact and extremely flexible; it uses a number of interchangeable furnaces with horizontal movement to provide easy access to the sample holder.



DIL 402 C (1600 °C model)

Measuring System
The high resolution (LVDT, 1.25 nm/digit) displacement transducer is employed in a newly designed Vacodil® (Invar) measuring system. The maximum measuring range is 5000 µm. The system yields extremely low drift and therefore measurements with unmatched repeatability and accuracy.

Automatic Zeroing
The pushrod is motorized and 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.

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 Control (STC) unit ensures excellent sample temperature control.

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

Adjustable Contact Pressure
In the DIL 402 C, the contact force of the pushrod can be adjusted between 15 cN and 45 cN. Smooth detection of sample dimension changes is guaranteed by an almost frictionless ball-bearing design supporting the pushrod.

Thermostatic Control
To ensure that there is no thermal influence from the furnace or from changes in room temperature, the support of the transducer is kept at constant temperature using an accurate thermostat. This guarantees reproducible measurements even in the most sensitive measuring range.

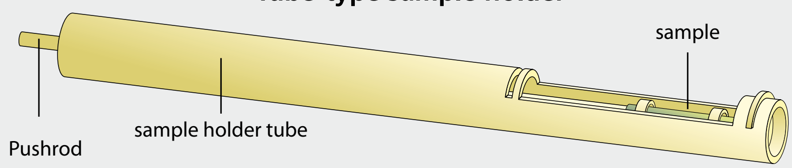
Atmosphere Control
The DIL 402 C is vacuum-tight by design, allowing careful control of the atmosphere and pure gas conditions. This feature is very useful for samples prone to oxidation, as the system can be evacuated and back-filled with a non-reactive gas. Of course, a static or dynamic reactive gas atmosphere can also be used.

DIL 402 C - Hardware

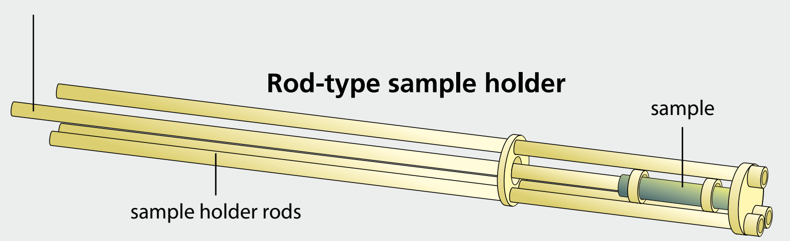
Tube-type Sample Carrier
The tube-type sample carrier is standard for the DIL 402 C. The sample is placed within the tube. Supports can be used for centering and to prevent direct contact between sample and carrier (sticking). The sample carriers are available in fused silica (max. 1100 °C), alumina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

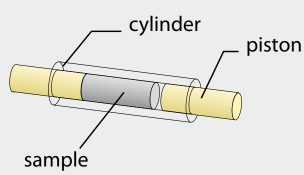
Rod-type Sample Carrier
The fused silica or alumina tube-type sample carriers can optionally be replaced with rod-type sample carriers for ideal heat transfer to the sample. They consist of three support rods, leaving the sample open to the heat source and the gas atmosphere. As a result, the accuracy is increased.

Tube-type sample holder



Rod-type sample holder





Sample container for pastes, powders and molten metals

Sample Containers
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. Protective sleeves for the sample are also available if reaction with the sample carrier is expected (Mo, BN, AIN, graphite).

High Vacuum
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 turbomolecular pump system is available.

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 a FT-IR via a transfer line. Outgasing of additives, organic binders and decomposition products can then be studied.

Large Sample Size
The maximum sample diameter for the standard sample holders is 12 mm. A special fused silica sample holder is available for diameters up to 19 mm.

Furnaces	Sample Holders	Thermocouples	Sample Dimensions	Atmosphere
low-temperature -180 °C to 500 °C	fused silica (tube and rod type)	type E	∅ 12 mm (max) length: 0 mm to 50 mm	inert, oxy., red., vac.
medium-temperature RT to 1100 °C	fused silica (tube and rod type)	types E, S	∅ 12 mm (max) length: 0 mm to 25 mm	inert, oxy., red., vac.
high-temperature RT to 1600 °C	fused silica, alumina (tube and rod type)	types E, S	∅ 12 mm (max) length: 0 mm to 25 mm	inert, oxy., red., vac.
maximum-temperature RT to 2000 °C	fused silica, alumina, graphite* (*only tube type)	types S, W	∅ 12 mm (max) length: 0 mm to 25 mm	oxy. (to 1680 °C), inert, red., vac.

Other NETZSCH Dilatometers	DIL 402 PC:	RT to 1600 °C
	DIL 402 ED/Cryo:	-260 °C to 50 °C
	DIL 402 E:	RT to 2800 °C
	DIL 402 CD:	differential or double sample arrangement for temperatures uo to 2000 °C

Software

The DIL 402 C runs under a 32-bit Windows® software package which includes everything necessary to carry out a measurement and evaluate the data produced.

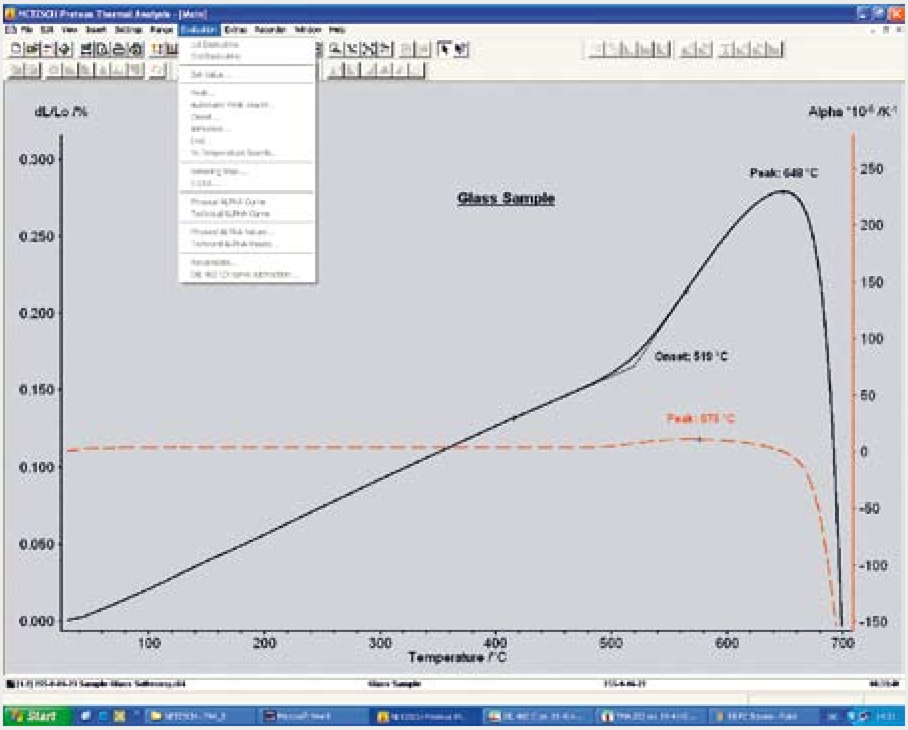
This software package is based on the experience of our Applications Laboratories and the ideas of countless customers over many years. The easy-to-understand menus and automated routines allow complicated analyses with an extremely user-friendly tool.

Standard Software Features:

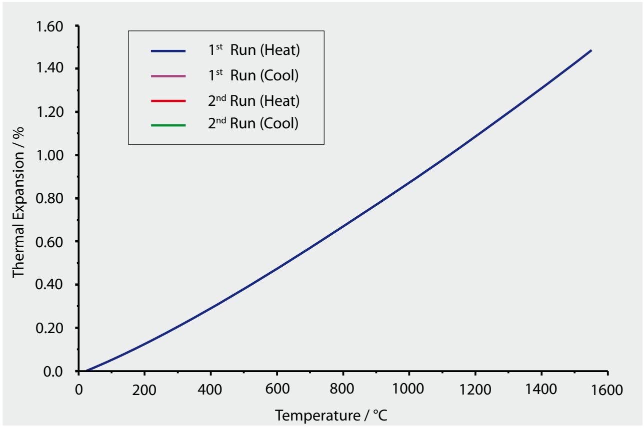
- for Windows® XP and Vista®-(Enterprise, Business) operating systems
- multi-tasking: simultaneous measurement and evaluation
- multi-moduling: operation of up to 4 different instruments with one computer
- combined analysis: comparison and/or evaluation of DTA/DSC, TG and DIL measurements in one plot (up to 32 measurements)
- labeling: input and free placement of text elements
- free scaling of text elements and evaluation results
- calculation of 1st and 2nd derivative
- selectable scaling
- graphic and data export
- selectable colors and line types
- storage and restoration of analysis with result at any stage
- context-sensitive help system
- temperature calibration
- zoom function

DIL Features

- various correction options:
  - sample holder expansion can be corrected with either a calibration measurement or a sample holder correction
  - offset correction
- characteristic temperatures: semi-automatic routines for determination of onset, peak and end temperatures
- glass transitions and softening points:
  - evaluations conform to DIN (German standards)
  - automatic softening point detection
- expansion coefficients: graphic or tabular presentation of technical and physical expansion coefficients
- analysis of sintering steps: automatic determination of the shrinkage during a sintering step
- Rate Controlled Sintering (RCS) software: optional software for measurements under RCS conditions in 3 different modes: start/stop, stepwise isothermal, dynamic heating rate

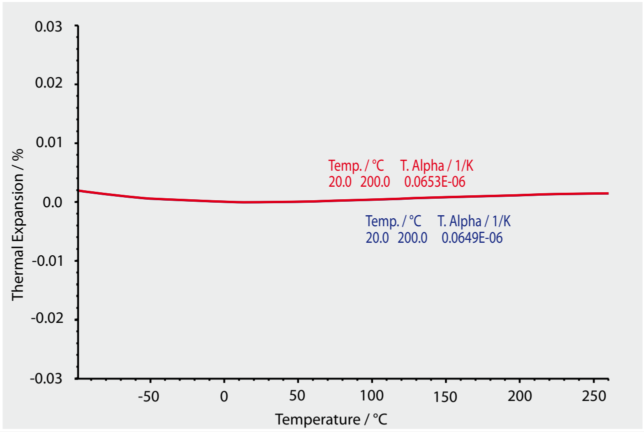


Performance



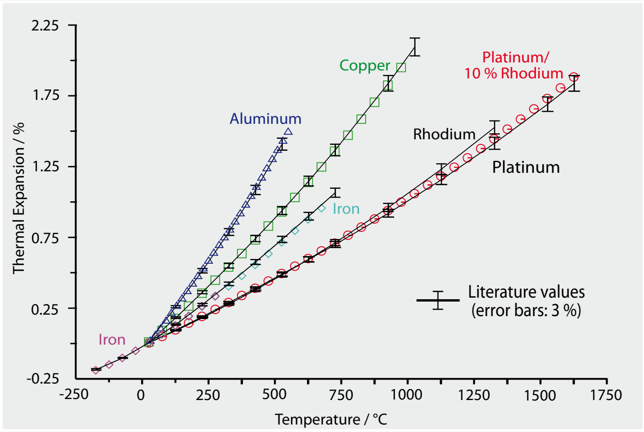
Unmatched Reproducibility

A sapphire sample was measured twice in 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 generally less than 0.3 %.



Excellent CTE Repeatability

A glass ceramic was measured twice between -100 °C and 260 °C using the low-temperature furnace. The heating rate was 3 K/min. A helium atmosphere was employed for the tests. The repeatability of the coefficient of thermal expansion between 20 °C and 200 °C was better than 1×10^{-9} 1/K. The repeatability of the raw data was therefore within the range of a few nanometers.



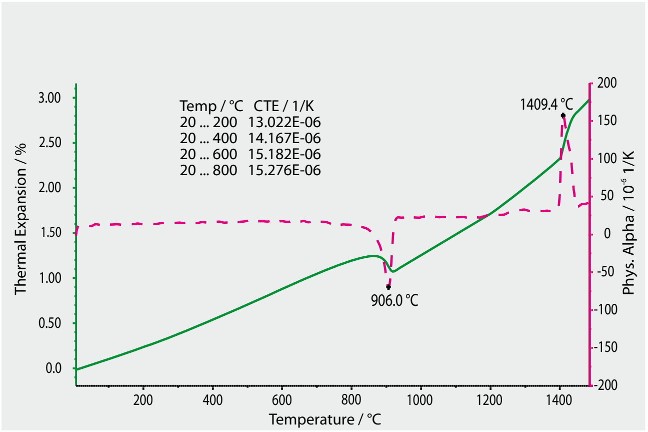
Outstanding Accuracy

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.

General Applications

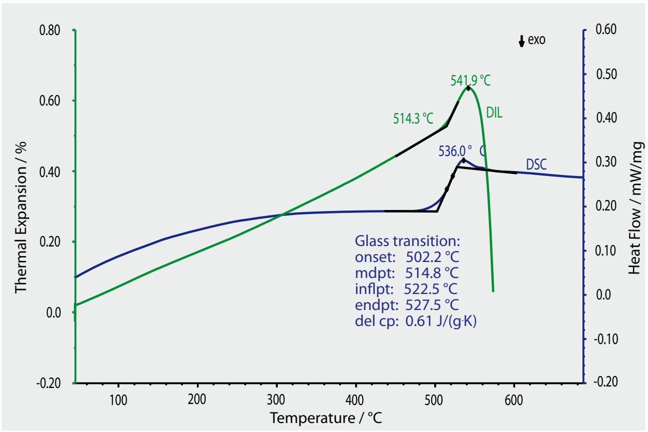
Iron

This figure depicts the linear thermal expansion and physical coefficient of thermal expansion (physical CTE) of iron. The sample was measured at a heating rate of 5 K/min in a helium atmosphere. At 906 °C (peak temperature in the physical alpha), a shrinkage step was detected. This is due to a change in the lattice structure (bcc→fcc). Another change in the lattice structure (fcc→bcc) was detected at 1409 °C. The deviation between the measured and literature transition temperatures is due to a small impurity content.



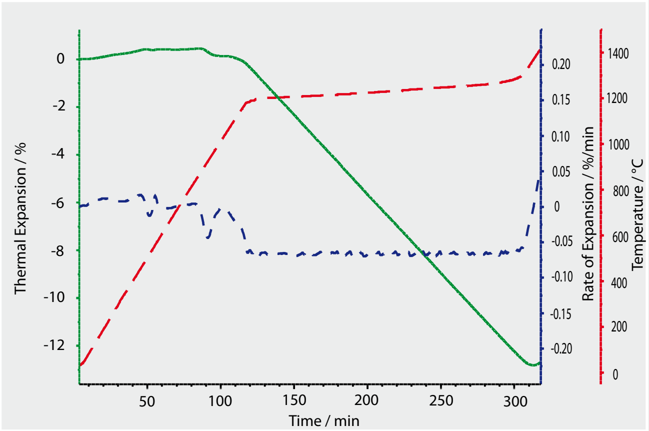
Glass

A glass sample was measured with the DIL 402 C and DSC 404 C *Pegasus*® under similar conditions. The DSC results clearly depict a step in the heat-flow rate due to the glass transition. The glass transition temperature was detected at 515 °C (mid point). In the dilatometer, the glass transition temperature was detected at 514 °C. The softening point was at 542 °C. The thermal expansion test was, of course, switched off automatically by the softening point detection in order to protect the system from contamination.

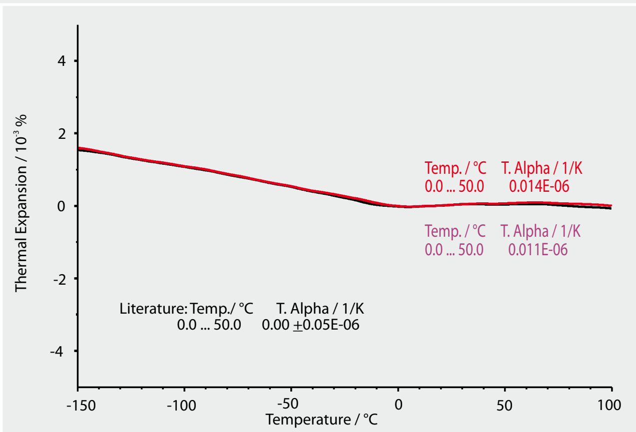


Alumina green body

An alumina green body was tested with the DIL 402 C employing the NETZSCH Rate Controlled Sintering (RCS) software. The measurement was carried out at a heating rate of 10 K/min. The start/stop mode of the RCS software was used. The threshold value was 10 µm/min (0.064 %/min). The heating rate was reduced by RCS during sintering to achieve a constant shrinkage rate. The influence of additives (e.g. organic binder, clays) was measured up to 1150 °C. The main sintering step occurred between 1150 °C and 1350 °C.

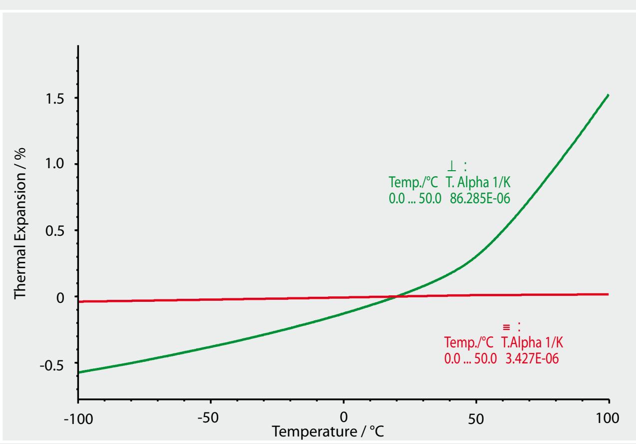


Low-Temperature Applications



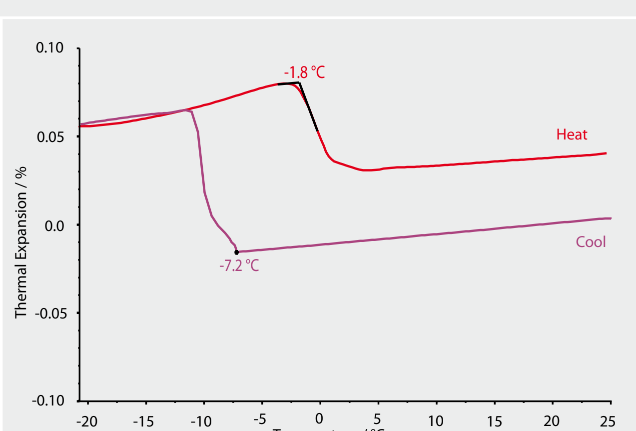
Glass Ceramic - Zerodur

Zerodur is a glass ceramic produced by Schott Glas in Mainz, 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.) Upon freezing, the sample length increases by approximately 0.08 %. Upon heating, the ice begins melting at -2 °C, resulting in shrinkage of the sample. The irreversible length change of the sample after cyclic cooling and heating may be due, in part, to cracks. Of course, such effects will reduce the life time of the roof tile in many climates.

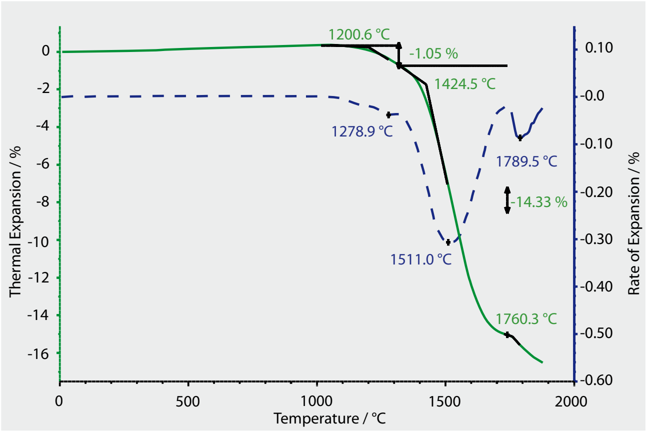
High-Temperature Applications - The 2000 °C Furnace

The DIL 402 C can be equipped with an innovative graphite furnace. In this furnace, the graphite heating element is designed such that - in spite of its compactness - it guarantees a very homogeneous temperature profile over the sample, even at high temperatures. The water-cooled furnace allows measurements between room temperature and 2000 °C in an inert atmosphere or vacuum.
When equipped with an alumina protective tube and sample carrier, measurements can be carried out up to 1680 °C under oxidizing conditions.

DIL 402 C/7 with 2000 °C furnace (cut-away view)

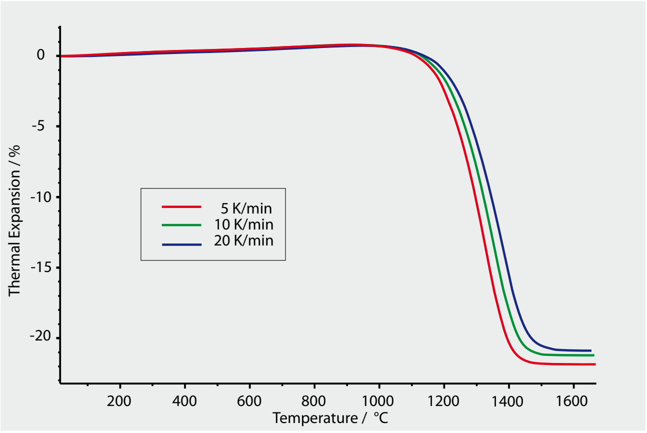
Silicon nitride

Because of its excellent thermal and mechanical properties, silicon nitride is used more and more for 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 figure 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 the sintering additives. The main shrinkage step occurred at 1424 °C (extrapolated onset). The effect above 1760 °C is most probably due to evaporation of additives.



Zirconia green bodies

Yttrium-stabilized zirconia green bodies were measured between room temperature and 1680 °C at 5, 10 and 20 K/min. The measurements were carried out in air using the 2000 °C furnace equipped with the Al_2O_3 sample carrier and pushrod. The results clearly show a dependence between heating rate and shrinkage. That is, higher shrinkage (densification) is achieved at lower heating rates. These results, of course, yield fruitful information for the optimization of the production process for such ceramics.

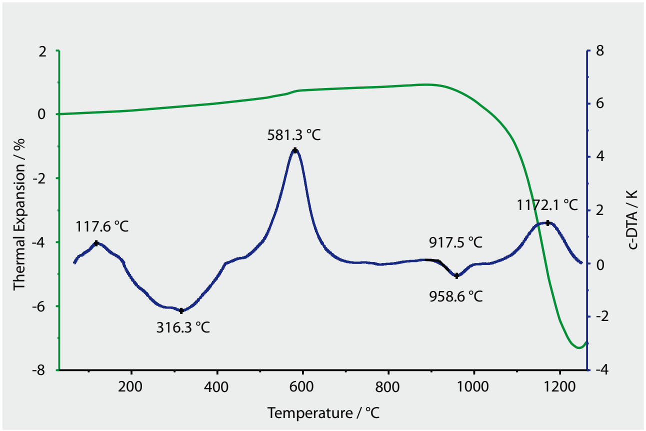


c-*DTA*® Feature

Dilatometry yields powerful infomation regarding the thermal expansion and shrinkage of materials during thermal treatment. On the other hand, interpretation of transitions occurring in the sample is sometimes difficult when using only thermal expansion data. The c-*DTA*® feature for the DIL 402 C is an additional tool for more detailed insight into material behavior. Differential Thermal Analysis (DTA) is a well-known tool for the characterization of transformation energetics (exo- and endothermal effects). Calculation of the c-*DTA*® signal on a thermal expansion curve yields information similar to the DTA method.

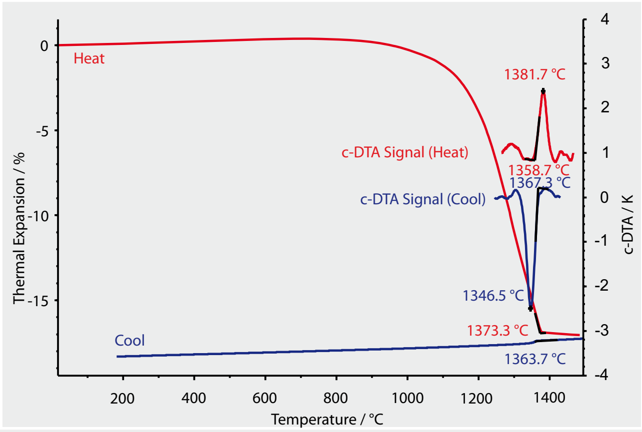
c-*DTA*® calculation is a mathematical routine based on the temperature measurement at the sample. Transitions connected with exo- or endothermal effects, of course, slightly influence the temperature change during dynamic heating or cooling. By comparing the measured temperature change of the sample with a theoretical one, these exo- or endothermal effects can be detected. If a calibration run on a standard sample is carried out prior to the sample run, this is also taken into consideration in the calculation process.

Application Examples:



Floor Tile

The thermal expansion and sintering of a floor tile during firing can easily be studied using the DIL 402 C. The additional c-*DTA*® feature allows more detailed insight into the thermal behavior of the material. The endothermal effect up to 150 °C is due to dehydration. The exothermal peak at 316 °C is due to the burnout of organic additives. Dehydroxilation of the clays was measured at 581 °C (peak temperature). The exothermal effect at 917 °C is due to a solid-state transition in the sample. The endothermal effect during sintering is most probably due to the formation of a liquid phase. (The c-*DTA*® curve was calculated stepwise.)



Tungsten Carbide + Cobalt

Tungsten carbide is a well-known hard metal used, among other things, for industrial cutting tools. In most cases, tungsten carbide parts are produced by a sintering process. This figure shows the thermal expansion measurement on a tungsten carbide green body during heating and cooling. It can be seen that sintering stops at 1373 °C. During cooling, a small step was detected at 1364 °C. This effect is most probably due to formation and solidification of a liquid phase. The c-*DTA*® calculation (endo- and exothermal effects) is another indication for this assumption.

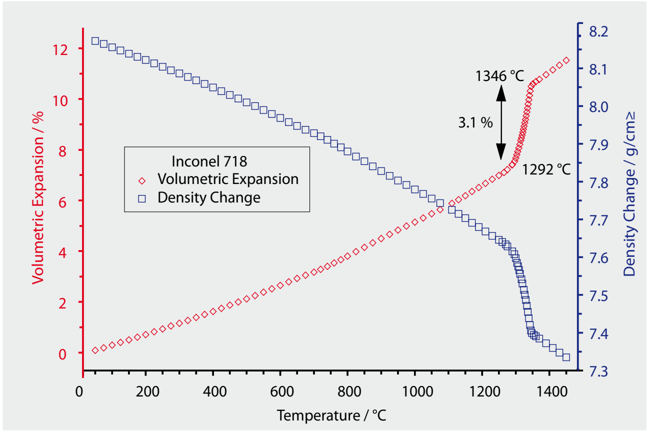
DIL 402 C - Specials

Volumetric expansion and bulk density of liquid metals

Dilatometers are generally used for measurement of solids. However, 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 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 the test. The influence of the container on the result was corrected by the NETZSCH Density 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 volumetric expansion were known, it was possible to calculate density versus temperature.

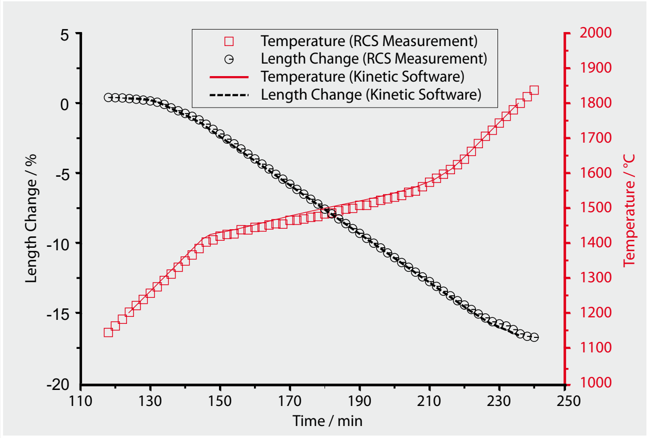


Advanced analysis and optimization of sintering processes

Characterization of the sintering behavior of ceramic or metal green bodies is a well-known application for pushrod dilatometers. Of course, temperature profiles for optimum sintering conditions can be measured employing the Rate Controlled Sintering (RCS) software. Combination of the DIL 402 C with the NETZSCH Thermokinetic Software package allows a more detailed analysis of sintering processes and offline optimization of the temperature profiles (e. g. calculation of temperature profiles for rate controlled sintering). The basis for this consists of measurements at different constant heating rates. These measurements are analyzed employing the thermokinetic software package. The result is in most cases a multiple-step formal-kinetic description of the test results. Using the description, it is possible to calculate the sintering behavior under different temperature profiles or to calculate the temperature profile for a constant shrinkage rate.

Silicon nitride

Silicon nitride green bodies were tested at heating rates of 5, 10 and 20 K/min between room temperature and 1900 °C. The results were described using a 4-step kinetic model in the NETZSCH Thermokinetic software. Based on this model, the temperature profile for constant shrinkage (0.174 %/min) was calculated. The figure shows a comparison of a real RCS measurement on a silicon nitride green body to the prediction of the thermokinetic software. The excellent agreement of the two results can clearly be seen and confirms the reliability of the new method.



The NETZSCH Thermophysical Properties Series

Knowledge of the thermophysical properties of materials is critical to material development and design in every modern industry. Accurate and efficient measurement of these properties, however, requires state-of-the-art instrumentation such as that produced by NETZSCH.

The DSC 404 ***F1/F3*** *Pegasus*®, LFA 427, DIL 402 C, STA 449 ***F1/F3*** *Jupiter*®, TCT 426 and TCT 416 form the core of the NETZSCH Thermophysical Properties Series instruments. Properties such as thermal diffusivity, thermal conductivity, specific heat, transformation enthalpies and temperatures, linear and volumetric thermal expansion, bulk density, mass change, etc. can be quickly and accurately determined using these instruments.

NBG · DIL 402 C · E · 500 · 1009 · LH · Technical changes reserved.

Leading Thermal Analysis.

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