NETZSCH

Proven Excellence.

Dilatometer Series
DIL 402 *Expedis Classic*

Method, Instrument, Applications

Analyzing & Testing

Dilatometry

NETZSCH dilatometers yield precise information on the expansion or shrinkage during thermal treatment providing insights into the behavior of ceramics, glasses and building materials. Specifically the knowledge of binder burnout during the firing process, sintering behavior, and influence of additives in the sintering process is required when producing ceramic materials. Even the development of glazes, e.g., for porcelain products, demands precise knowledge of the dimensional changes of the products in contact during firing.

Changes in the composition of glasses can be simply and quickly determined by measurement of the expansion coefficients or determination of the glass transition temperature. In various cases, it is important to match the thermal expansion behavior of different glasses that are in contact with one another in order to avoid stresses and possible cracking.

Moisture and phase transitions influence the expansion and shrinkage behavior of building materials, e.g., concrete. These can significantly influence the static reliability and durability of the systems they are used in.

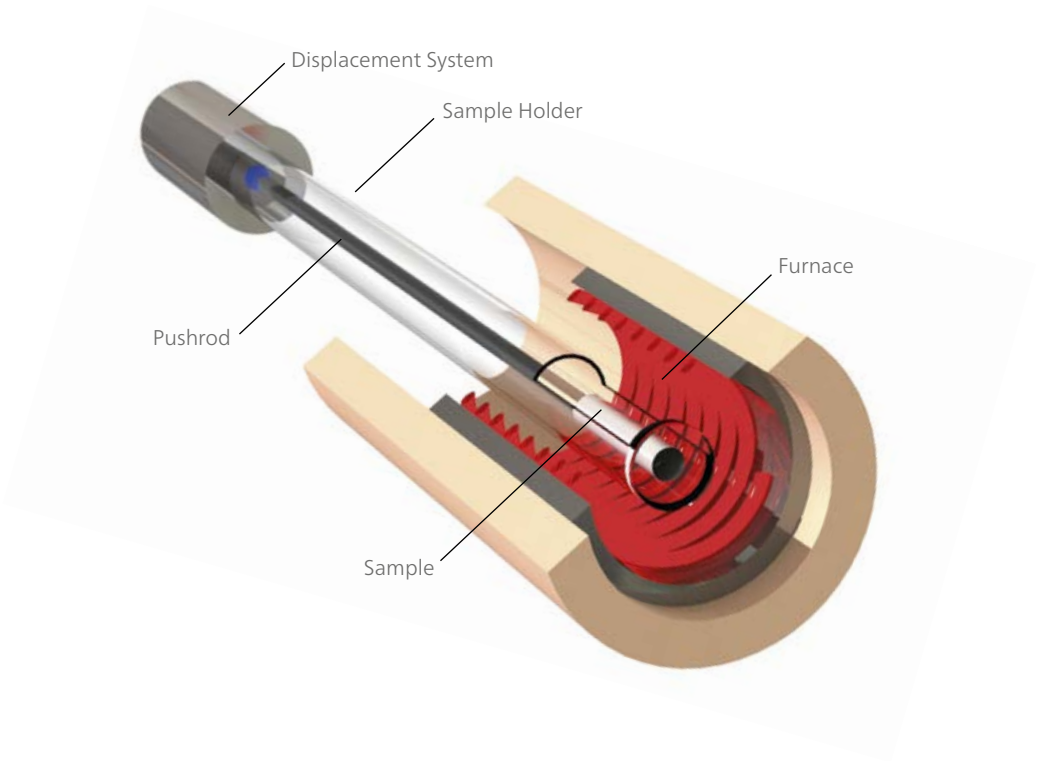
The investigation of dimensional changes such as expansion, shrinkage including volume changes is provided by dilatometry. For decades, this method has been successfully established at industrial and research centers. All NETZSCH dilatometers are based on, e.g., DIN EN 821, DIN 51045, ASTM E831, ASTM E228.

The Method for Determination of Dimensional Changes

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 (α).

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

α	coefficient of expansion
L_0	initial sample length
ΔT	change in temperature
Δl	change in length



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

For preparing a dilatometer measurement, a rod-shaped sample, typically several cm long, is inserted into a sample holder and brought into contact with the pushrod. After closing the furnace, the experiment can be started.

The thermal expansion of the sample during heating, cooling or under isothermal conditions is detected by the displacement system which the pushrod is connected to.

Information from DIL measurements:

- Linear thermal expansion
- Coefficient of thermal expansion (CTE)
- Volumetric expansion
- Shrinkage steps
- Softening point
- Glass transition temperature
- Phase transitions
- Sintering temperature and step
- Density change
- Influence of additives and raw materials
- Decomposition temperature of e.g., organic binders
- Anisotropic behavior
- Optimization of firing process
- Caloric effects by using c-*DTA*®

User-Optimized Environment

EASE OF USE

Major simplification begins with preparation of the measurement by using method-based test routines predefined by the operator. The *MultiTouch* feature places the sample into the optimum position using a unique, tail-like motion. The initial sample length is then automatically determined at a predefined contact force. Exchanging the furnace is simple and requires no detailed experience. Only a few clicks are needed to start the measurement.

ALL-IN-ONE DESIGN

DIL *Expedis Classic* is available as a single or dual/differential system. In both versions, the all-in-one design incorporates all hardware components which are usually necessary for precise dilatometer measurements. There is no inconvenient cable tangle nor is an external chiller required.

LONG LIFE CYCLE

Expedis Classic supports your smooth production flow by maintenance-free operation, low work load, long life cycle, simplified and safe operation. The system's optimized design also includes effortless exchanging of the sample holder system with no risk of distortion.

BEST SAMPLE CONDITIONS

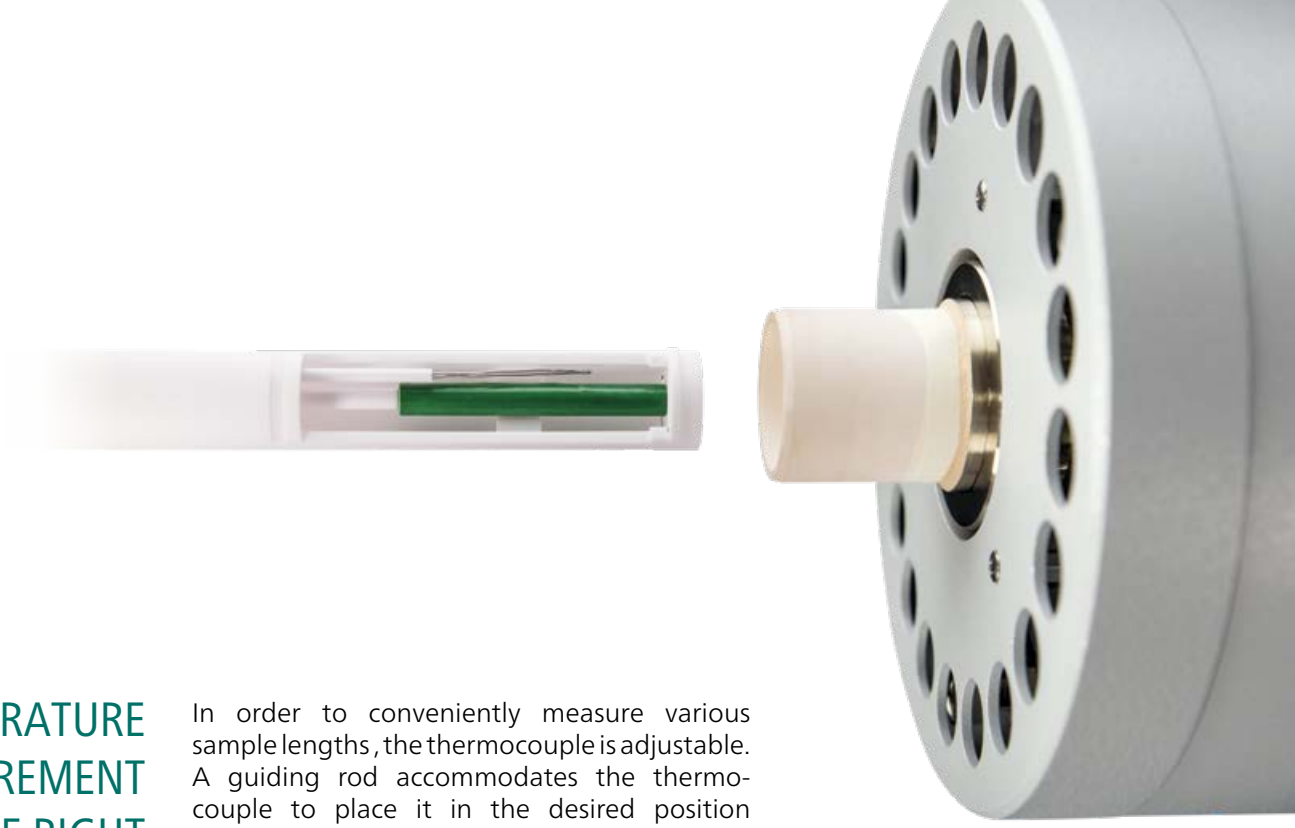
Defined atmosphere by gas-tight design and superior insulation are prerequisites for precise measurement results. Such measurement conditions are realized by the gas-tight design and integrated gas flow device. The gases can be directed through the measuring cell and sample compartment. In addition, insulation of the sample compartment reduces influences of temperature fluctuations on the measurement results.

MODULAR CONCEPT
SINGLE OR DOUBLE DILATOMETER
HIGH SAMPLE THROUGHPUT
FLEXIBILITY
FAST START OF MEASUREMENT
WIDE TEMPERATURE RANGE
TUBE TYPE SAMPLE HOLDERS
ROD TYPE SAMPLE HOLDERS
FURNACE VARIETY
EXCHANGEABLE FURNACES
CUSHIONING OF THE FURNACE
ROBUST
NANOEYE
AUTOMATIC SAMPLE LENGTH DETERMINATION
PERFECT LINEARITY
EASE OF USE
HIGH RESOLUTION
ALL-IN-ONE DESIGN
CONSTANT RESOLUTION
MULTITOUCH
LOW COST OF OPERATION
HIGH REPRODUCIBILITY
MAINTENANCE FREE
HIGH PRECISION
VARIABLE CONTACT FORCE
GAS-TIGHT
FRAGILE SAMPLES
SOFT SAMPLES
INNOVATIVE FORCE CONTROL
INSULATED MEASUREMENT CELL
NO CHILLER
PRECISE CONTACT FORCE
SLIDING THERMOCOUPLE
LONG-TERM MEASUREMENTS
DEMANDING APPLICATIONS
DEFINED ATMOSPHERE
FAST & EASY
SOLID SAMPLES
SIMPLE

Dilatometry Redefined

CONTROLLED CONTACT FORCE

The controlled contact force allows the operator to measure small, delicate, fragile or foamed samples without risk of breakage and any non-reproducible deformation. The contact pressure over the entire measurement time is kept constant independent of the expansion or shrinkage of the sample. Sliding and rolling friction, stick-slip effects, etc. in the measurement system will now also be avoided.



TEMPERATURE MEASUREMENT AT THE RIGHT SPOT

In order to conveniently measure various sample lengths , the thermocouple is adjustable. A guiding rod accommodates the thermocouple to place it in the desired position without bending.

NanoEye OBSERVES YOUR SAMPLE

The new, pioneering *NanoEye* displacement system features perfect linearity and maximum resolution over the entire measuring range. The heart of the *NanoEye* is an opto-electronic sensor able to encode a position and convert it into a digital signal. Using a linear encoder has the advantage that resolution, accuracy and linearity remain stable over the entire measurement and temperature range. In addition, *NanoEye* allows for automatic determination of the sample length.

Proteus® Software

Best Practice for Measurement and Evaluation

The unique *Proteus*® dilatometer software offers everything a user could ever want and need: It runs smoothly, provides reliable results, and it is fast and efficient. It provides a large range of functions, but – at the same time – offers a clearly-arranged user interface. Additionally, it is intuitive and thus easy to learn.

But … that's not all. There are some more options inside which impress even the most experienced operators – particularly the *Density Determination*, the patented c-*DTA*®, Rate-Controlled-Sintering and the new, innovative *Identify* software features.

Special Features of the *Proteus*® Software for DIL 402 *Expedis Classic* at a Glance	
Software controlled force adjustment (incl. constant force)	
*Density Determination**	
c-*DTA*® *	for temperature calibration or determination of caloric effects
RCS*	Rate-Controlled Sintering
*Identify**	identification of unknown $\Delta L/L_0$ curves through database comparison

Advanced Software (for extended evaluation of the measuring data)
*Thermokinetics**
*PeakSeparation** (for processing the 1st derivative)

* optional

Density Determination

This program add-on allows determination of the density change of samples with varying consistency, i.e., solids, viscous materials such as pastes, liquids or melts as well as the volumetric expansion of isotropic materials.

Patented* c-*DTA*®

The c-*DTA*® signal gives the opportunity for simultaneous analysis of length changes and endothermal/exothermal effects. It can also be used for temperature calibration.

* DE102013100686

Identify

Built-In Thermal Analysis Expertise

The innovative *Identify* software extension for the identification and interpretation of DIL measurements includes several NETZSCH libraries with hundreds of entries from the ceramic, inorganic, metal, alloy, and polymer or organic fields. Additionally, user-specific libraries can be created. They can be shared with other users within a computer network.
Identify allows the identification of unknown samples from the measured curve shape. This will also open up the opportunity to compare known samples against a variety of other samples, enabling one to make a statement about prinicipal material behavior. All measurements can be stored in the extensive database and are always available for identification or quality comparison of future measurements.

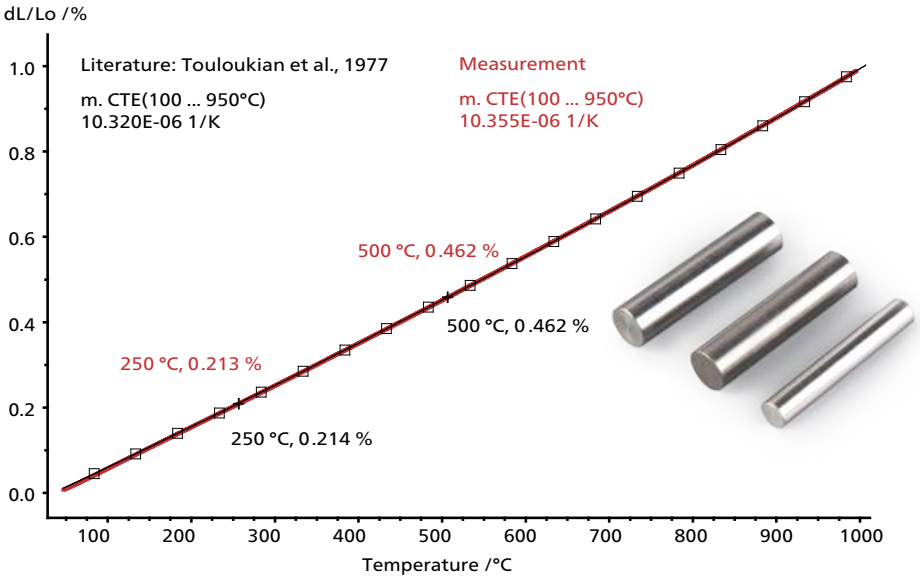
Identify provides all information with one single click

Identification
of unknown measurement curves

Quality Control
via agreement between the current measurement and selected database entries

Archiving Functionality
for present measurements and existing database entries

Performance and Applications



Platinum measurement in comparison to literature values. Measurement conditions: heating rate 5 K/min, dynamic nitrogen atmosphere (20 ml/min).

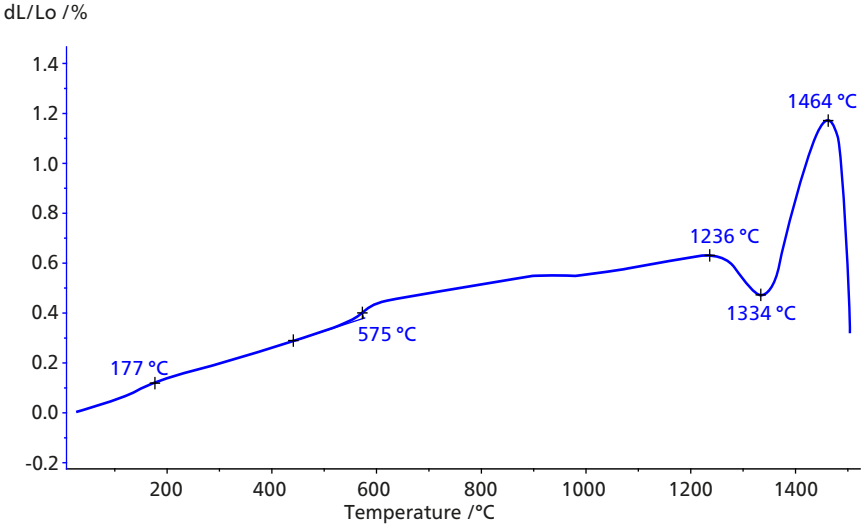
High Accuracy

This plot exhibits the measurement (red curve) on a reference material made of platinum between room temperature and 990°C. The black curve corresponds to literature values. The deviation of the determined mean CTE value between 100°C and 950°C is less than 4E-08 1/K in comparison to literature data [1].
The software includes expansion data of common reference materials. For convenient comparison, these tables can be loaded as curves into the evaluation window of the dilatometer soft- ware allowing for graphical illustration of the accuracy.

[1] Thermophysical Properties of Matter, Touloukian et al. (1977) Vol. 12

Expansion Behavior of a Refractory

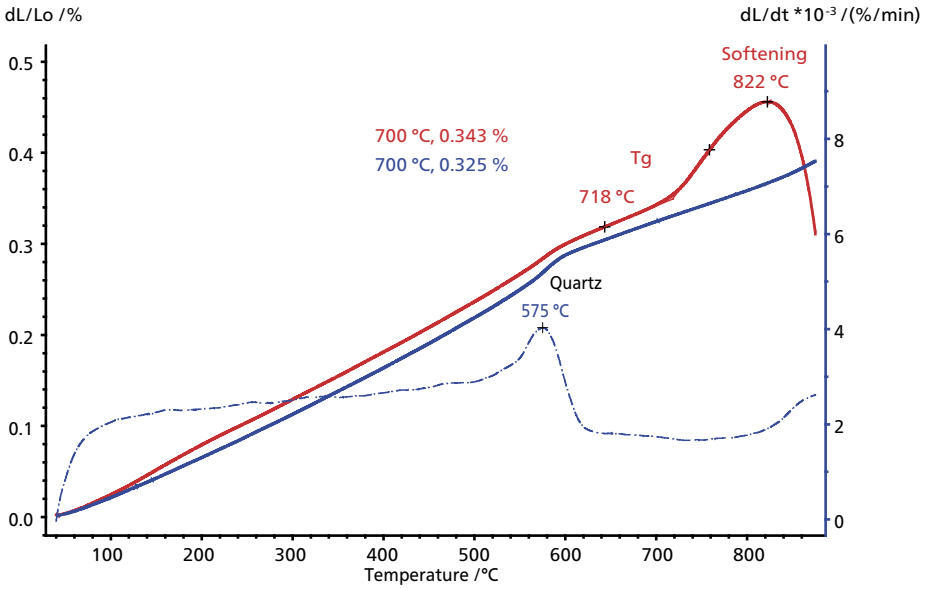
One of the important criteria in assessing refractory-materials from the thermal shock resistance point-of-view is their thermal expansion together with their high-temperature resistance. This example shows the thermal expansion of such a material. A phase transition of the tridymite content in the refractory was detected at 177°C. This transition is followed by the α-ß transformation of the free quartz at 575°C (onset temperature). Between 1230°C and 1334°C, a phase transition can be observed. After a short expansion step, the material begins to soften at a peak temperature of 1464°C.



Phase transitions and softening of a refractory material between room temerature and 1500°C. Measurement conditions: heating rate 5 K/min, air atmosphere.

Thermal Expansion Mismatch

Poor glaze/body fit is the main cause of crazing (spider web pattern of cracks penetrating the glaze). This effect is caused by, e.g., thermal expansion mismatch which can be avoided by adjusting the thermal expansion behavior of the body and the glaze. This plot shows the expansion behavior of the glaze (red curve) compared to that of the body to which it should be applied. At 700°C – shortly before the glass transition temperature of the glaze at 718°C – the difference in expansion amounts to 0.02%. Softening of the glaze occurs at 822°C. The higher expansion of the glaze could lead to unwanted tensile stress during cooling which is proportional to the thermal expansion.



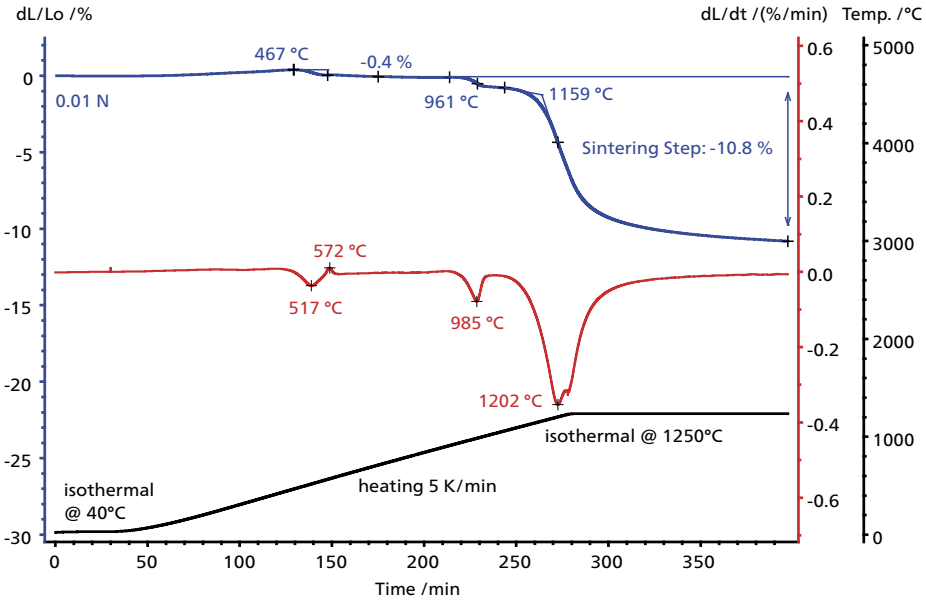
Comparison of the thermal expansion of a glaze and the body on which it should be held. α → β-transition of quartz is detected at 575°C, shown here in the 1st derivate of the body (blue dotted curve). Measurement condition: heating rate 5 K/min, air atmosphere.

Applications

Understanding of Sintering Behavior

Porcelain is a ceramic material with variable composition mainly containing kaolinite, feldspar, and quartz. The formation of glass and mullite within the fired body at high temperatures (>1200°C) is responsible for the porcelain's toughness, strength, and translucence.

During heating of the porcelain green body, dehydration of the kaolinite occurs in the temperature range between 450°C and 570°C which leads to the formation of metakaolinite (peak at 467°C in the thermal expansion (blue curve), related to the peak at 517°C in the 1st derivative (red curve)). The temperature range indicates the release of chemically bound water of the clay crystal structure which involves shrinkage of approximately 0.4%. The peak at 572°C in the 1st derivative results from the α→β transition of quartz. A further effect can be observed at 961°C (blue curve), related to the peak at 985°C in the 1st derivative (red)) which can be attributed to the structural collapse of metakaolinite and the formation of γ-Al_2O_3 [1]. With the complete melting of feldspar and the formation of mullite, two-step sintering starts above 1159°C. The total shrinkage was determined to 10%.

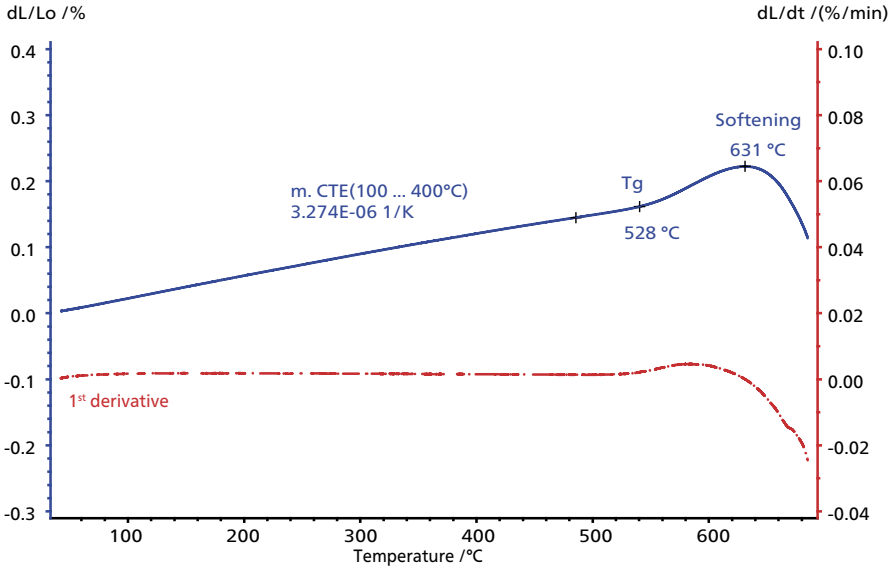


Porcelain green body. Measurement conditions: RT up to 1250°C, heating rate 5 K/min, dynamic air atmosphere (20 ml/min), contact force of the push rod 0.01 N

[1] Classic and Advanced Ceramics: From Fundamentals to Applications, Robert B. Heimann, 2010 WILEY-VCH Verlag GmbH & Co. KGaA, Weinheim

Determination of Quality Parameters in Glass Production

Borosilicate glasses are characterized by low thermal expansion coefficients making them resistant to thermal shock. In addition, these glasses have excellent optical, chemical and mechanical properties which allow for high quality products such as implantable medical devices and devices used in space exploration. This plot shows the thermal expansion of a borosilicate glass between room temperature and 700°C. The glass transition was determined at 528°C (extrapolated onset), softening of the glass occurred at 631°C.



Thermal expansion curve and 1st derivative of a borosilicate glass. Measurement conditions: Heating rate 5 K/min, air atmosphere.

Technical Specifications

DIL 402 *Expedis Classic*	
Design	Pushrod dilatometer, single or dual system
Furnaces	▪ Fused silica: RT to 1150°C ▪ SiC: RT to 1600°C (optional furnace for fast cooling)
Heating rates	0.001 … 50 K/min
Cooling systems	Air compressor or connection set (ballistic cooling; only for optional SiC-furnace for fast cooling)
Sample holder systems	Interchangeable, made of fused silica and alumina, in two versions ▪ Single system (one pushrod) ▪ System with two pushrods usable in dual or differential mode ▪ Al_2O_3 tension sample holder*
Sample dimensions	Sample length max.: 52 mm ▪ Ø 12 mm standard (optional Ø 19 mm max.) ▪ Ø 8 mm in dual sample holder system
Automatic sample length determination	Yes
Displacement system	*NanoEye*
Temperature accuracy	1 K
Temperature precision	0.1 K
Temperature resolution	0.001 K
Thermal stability (isothermal)	± 0.02 K
Temperature calibration	▪ Via displacement using metal references ▪ c-*DTA*® (optional, incl. endo/exothermal effects)
Measuring range	± 5000 µm
ΔL Resolution	2 nm (over the entire measuring range)
$\Delta L/L_0$ Repeatability	0.002 %, absolute value
$\Delta L/L_0$ Accuracy	0.003 %, absolute value
Force range (load at the sample)	0.01 N … 3 N (valid for compressive and tensile force depending on the sample holder)
Force resolution	0.001 mN
Gas atmosphere	Inert, oxidizing under static or dynamic conditions
Gas control	1-way, optional 3-way switch
Gas-tight	Yes
Software	Windows 7 32/64 bit Professional®, Windows 7 32/64 bit Enterprise®, Windows 7 32/64 bit Ultimate®, Windows 8.1 Pro® and Enterprise® Windows 10 Pro® and Enterprise®

* Please note, using the tension sample holder has an influence on the noise behavior.

All over the world, the name NETZSCH stands for comprehensive support and expert, reliable service, both before and after sale. Our qualified personnel from the technical service and application departments are always available for consultation. In special training programs tailored for you and your employees, you will learn to tap the full potential of your instrument.

To maintain and protect your investment, you will be accompanied by our experienced service team over the entire life span of your instrument.

Expertise in SERVICE

TECHNICAL SERVICE

- Maintenance and Repair
- Software Updates
- Exchange Service
- IQ/OQ Documents
- Calibration Service
- Spare Part Assistance
- Moving Service

TRAINING

- Training
- Comprehensive Instrument and Method Training

LABORATORY

- Application Service and Contract Testing

The NETZSCH Group is an owner-managed, international technology company with headquarters in Germany. The Business Units Analyzing & Testing, Grinding & Dispersing and Pumps & Systems represent customized solutions at the highest level. More than 3,700 employees in 36 countries and a worldwide sales and service network ensure customer proximity and competent service.

Our performance standards are high. We promise our customers Proven Excellence – exceptional performance in everything we do, proven time and again since 1873.

When it comes to Thermal Analysis, Calorimetry (adiabatic & reaction), the determination of Thermophysical Properties, Rheology and Fire Testing, 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.

Proven Excellence.

Headquarters
NETZSCH-Gerätebau GmbH
Wittelsbacherstraße 42
95100 Selb
Germany
Tel.: +49 9287 881-0
Fax: +49 9287 881 505
at@netzsch.com

India
NETZSCH Technologies
India Private Limited
No. 39, 2nd Street Spartan Nagar,
Mogappair, Chennai – 600 037
Tel.: +91 44 42965111
Fax: +91 44 42965115
at@netzsch.com

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