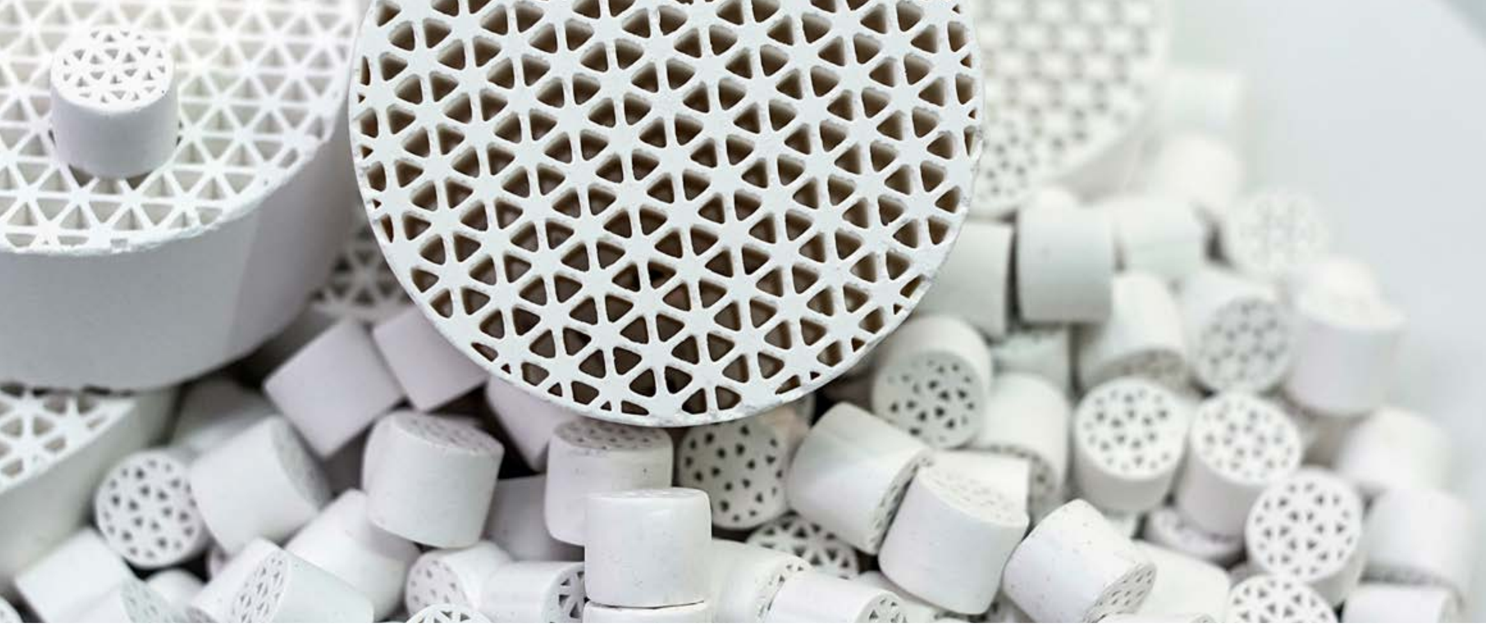
NETZSCH

Proven Excellence.

Dilatometry – DIL 502 *Expedis*® Series

Method, Instruments, Applications

Analyzing & Testing



Dilatometry

Dilatometry is a precise analytical technique that measures dimensional changes in materials as a function of temperature and/or time. It provides critical information on key material properties. These measurements are fundamental for both quality assurance and the advancement of materials research. Over the past decades, dilatometry has been firmly established as a reliable method in industrial and research settings. All NETZSCH dilatometers are based on such standards as DIN EN 821, DIN 51045, and ASTM E228.

Ceramics and Glass: Dilatometry provides insight into the sintering process and thermal behavior of these materials, which is crucial for their application in construction and electronics.

Metals and Alloys: The study of phase transitions in steel (e.g., martensite) and thermal expansion coefficients an be used to analyze temperature-time-length relationships, revealing microstructure changes and optimizing heat treatment.

Composites: Determining thermal expansion in composites is key to ensuring material compatibility – for example, in reinforced concrete, where steel and concrete must have closely matched expansion rates.

Polymers: Dilatometry is employed to ascertain glass transition temperatures and thermal expansion coefficients, facilitating a comprehensive understanding and optimization of polymer processing, design, and performance across a range of applications.

Refractories: Dilatometry reveals the thermal behavior of refractories – such as expansion, phase transitions, and sintering – by measuring dimensional changes under controlled temperatures.

Information from DIL Measurements

- Linear thermal expansion
- Coefficient of thermal expansion (CTE)
- Softening point
- Glass transition temperature
- Phase transitions
- Sintering temperature and shrinkage steps
- Influence of additives and raw materials
- Decomposition temperature of, for example, organic binders
- Anisotropic behavior
- Volumetric expansion
- Density change
- Caloric effects by using c-*DTA*®

The Method for Determination of Dimensional Changes

Pushrod dilatometry is a method for determining dimensional changes versus temperature and/or time while the specimen undergoes a controlled temperature program. The degree of expansion divided by the change in temperature is called the material's coefficient of expansion (ā).

$$\bar{a} = \frac{1}{L_0} \left(\frac{\Delta L}{\Delta T} \right)$$

$\bar{a}$	mean coefficient of expansion
L_0	initial specimen length
ΔT	change in temperature
ΔL	change in length

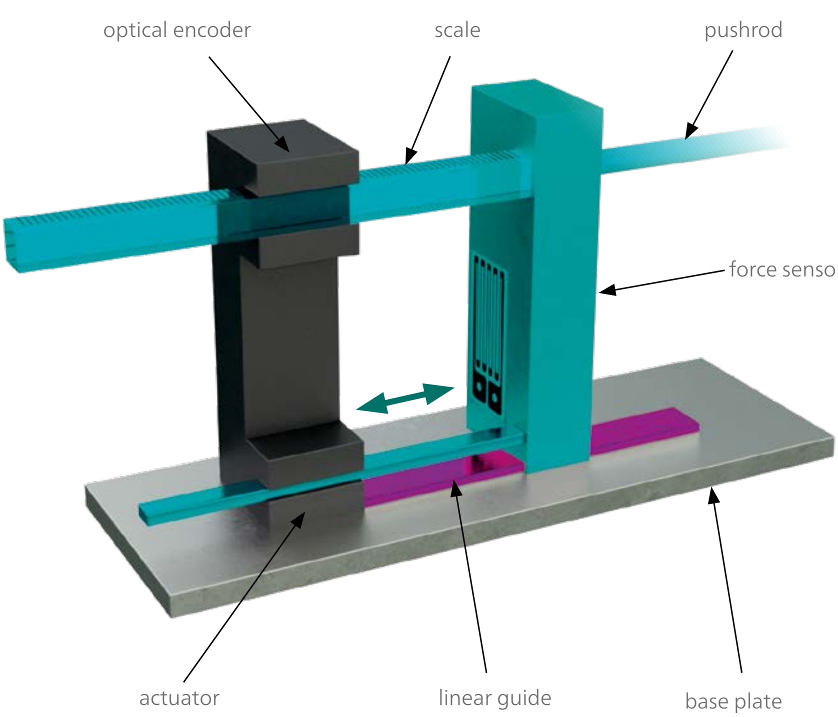
To prepare a dilatometer measurement, a specimen is inserted into a sample holder and subjected to a defined application of force by a pushrod. After closing the furnace, the experiment can be started.

The length change of the specimen during heating or cooling, or while under isothermal conditions, is detected by the displacement system to which the pushrod is connected.

DISPLACEMENT SYSTEM

Opto-Electronic Measuring System *NanoEye*

In traditional dilatometry, increasing resolution often reduces the measurement range and vice versa. The *NanoEye* system overcomes this limitation by offering high resolution alongside an unmatched measurement range. It achieves perfect linearity in thermal expansion measurements, surpassing conventional systems.



Schematic of the *NanoEye* measuring cell

The *NanoEye* consists of:

- An actuator which applies a controlled contact force and moves the pushrod to adjust to variable specimen lengths
- An elastic force sensor which detects the contact force
- An optical encoder (plus scale) which measures the initial specimen length and determines the length change of the specimen

Functional Principle

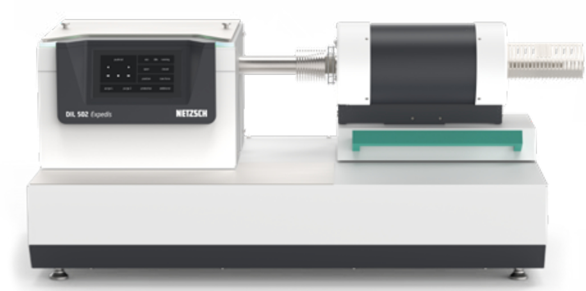
During a test run, when the specimen changes length, all green components in the graphics move with the help of a linear guide (marked in purple). The optical encoder determines the corresponding length change directly on the appropriate scale.

The DIL 502 *Expedis*® Series

Key Insights into the Expansion and Shrinkage of Ceramics, Glasses, Metals and Building Materials

Classic

- RT to 1600°C
- Furnaces: SiO_2, SiC*
- Resolution: 2 nm
- Measurement range: ± 5 mm
- Gas-tight



The Work Horse – Offering the Best Price/Performance Ratio

The DIL 502 *Expedis*® *Classic* facilitates industrial quality control and application through maintenance-free operation, thereby ensuring operational efficiency and reliability. The all-in-one design, which eliminates the need for supplementary chiller or power units, helps furnish a clutter-free work environment in high-pressure industrial settings. For high sample throughput, the *Classic* can be configured with a dual furnace and a dual measuring system. The robust design enables operation from ambient temperature to 1600°C with a resolution of up to 2 nm.

* Available in different versions. See page 10.

Select

- -180°C to 2000°C
- Furnaces: Copper, Steel, SiO_2, SiC*, Rh, Graphite
- Resolution: 1 nm
- Measurement range: ± 10 mm
- Vacuum-tight

The All-Rounder for Most Applications

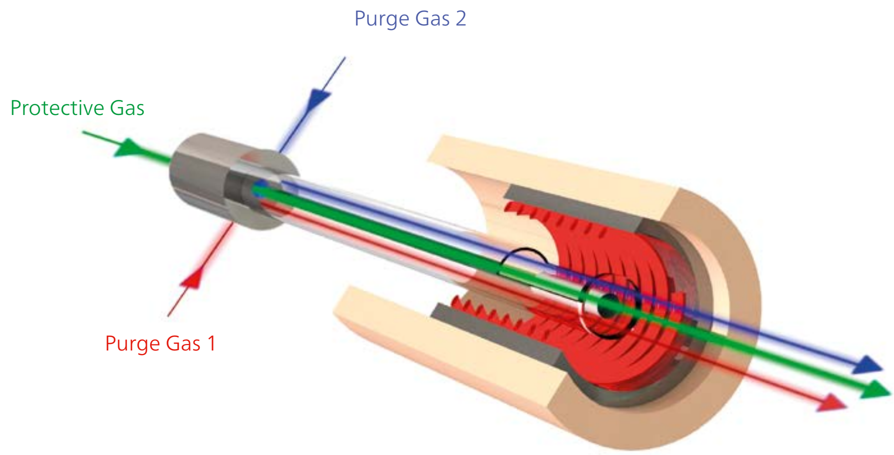
Perfect for sophisticated industrial research and contract laboratories, the *Select* model offers a flexible instrument setup. This instrument can operate at temperatures ranging from -180°C to 2000°C without the need to exchange furnaces when using the dual furnace design. Choose from seven different furnaces to meet your temperature needs, such as the copper furnace (-180°C to 500°C) or the graphite furnace (RT to 2000°C).

Supreme

- -180°C to 2800°C
- Furnaces: Copper, Steel, SiO_2, SiC*, Rh, Graphite*
- Resolution: 0.1 nm
- Measurement range: ± 25 mm
- Vacuum-tight

The Expert – Highest Level of Sophistication

The *Supreme* version is designed for high-end research and development and sophisticated industrial applications: The comprehensive, fully-equipped *Supreme* model allows for temperatures from -180°C to 2800°C in one instrument and features an exceptional resolution of up to 0.1 nm.



Schematic of the gas paths inside the instrument when using one protective gas and two purge gases

Separate Paths for Protective and Purge Gases

When using the three mass-flow controllers (4th is optional), the gas flow paths inside the instrument are divided: The protective gas is first directed through the measuring cell and subsequently enters the sample chamber, whereas the purge gas(es) is/are directly fed into the sample chamber.
All protective and purge gases leave the instrument together via the furnace exhaust. In the standard version, if only one gas switch or one MFC is integrated, the gas follows the same path as the protective gas.

COMBINES ADVANCED TECHNOLOGY WITH USER-FRIENDLY FEATURES

The DIL 502 *Expedis*® Series

TAILOR-MADE INSTRUMENT

Customize your setup with a single or dual furnace and/or measurement system. The dual furnace system allows for an impressive temperature range from -180°C to 2000°C (available in the DIL 502 *Expedis*® *Select* and *Supreme*).

SAMPLE HOLDER VERSATILITY

Diverse specimen geometries can be accommodated with single or dual sample holders and a sample holder for tension measurements.

EVOLVED GAS ANALYSIS AND VACUUM

The vacuum-tight designs of the DIL 502 *Expedis*® *Select* and *Supreme* permit connection to a QMS or FT-IR to study the outgassing of impurities, additives or degradation products. Optional evacuation systems allow for measurements under vacuum.

Automatic Specimen Length Detection

Say goodbye to the inconsistencies of manual measurements. The DIL 502 *Expedis®* automatically detects the initial length of a specimen before testing, ensuring accuracy under identical conditions.

MultiTouch Technology

Achieving stable specimen positioning is crucial for reliable results. The *MultiTouch* feature automatically places the specimen in the optimal position using a unique tail-like motion, enhancing measurement consistency.

Adjustable Thermocouple Positioning

Easily measure specimens of varying lengths with the adjustable, capillary-guided thermocouple – precise positioning without bending.

Bridging the Gap to TMA

The DIL 502 *Expedis®* series is the first horizontal dilatometer series on the market; it allows for force modulation and, by this means, bridges the gap between dilatometry and thermomechanical analysis (TMA) under oscillatory load.

AutoVac – Measurements Under Inert Atmospheres

The optionally available *AutoVac* allows for quick evacuation and gas re-fill for measurements in pure inert atmospheres, e.g, for oxidating materials.

High Specimen Throughput

To maximize efficiency and productivity, the optional dual furnace design, coupled with a dual measuring system, signifi-cantly increases measurement capacity.

FURNACE OPTIONS

Select from nine distinct furnace models* to precisely match your temperature requirements. Choose between manual and motorized furnace operation, optimizing workflow efficiency.

* depending on instrument version. See page 10.

UNMATCHED MEASUREMENT RANGE AND RESOLUTION

Experience the highly precise opto-electronic displacement system *NanoEye*, which delivers exceptional linearity and resolution over a wide measure-ment range to capture even the smallest dimensional changes.

OPTIMIZED EFFICIENCY FOR MORE SUSTAINABILITY

The combination of electronic thermal stabilization and the Eco/Idle Mode feature ensures peak performance while minimizing energy and gas consumption, thus allowing for significant savings. (See page 8).

Thermal Analysis Made More Sustainable

REDUCED ENERGY CONSUMPTION AND OPERATING COSTS

Idle/Eco Mode

General | **Schedule**

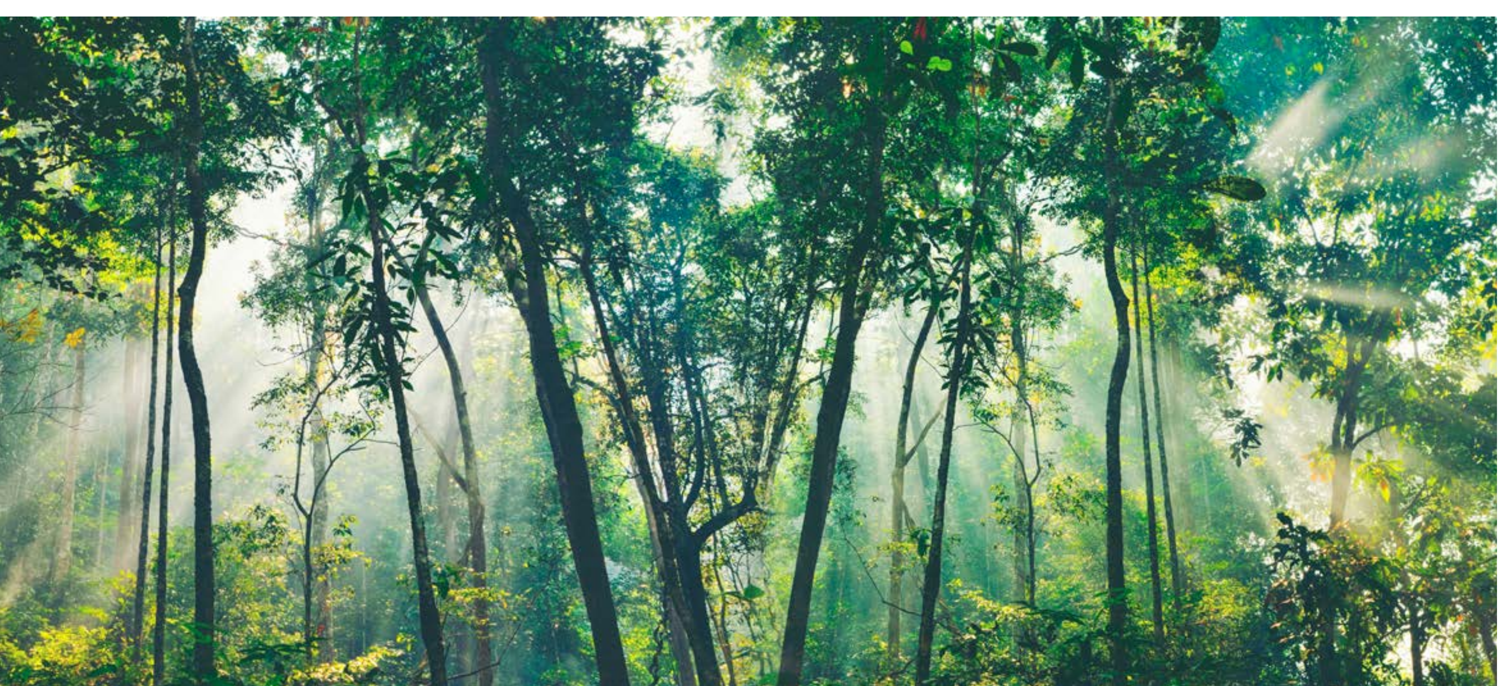
	Sunday	Monday	Tuesday	Wednesday	Thursday	Friday	Saturday
12:00 AM	ECO	ECO	ECO	ECO	ECO	ECO	ECO
1:00 AM	ECO	ECO	ECO	ECO	ECO	ECO	ECO
2:00 AM	ECO	ECO	ECO	ECO	ECO	ECO	ECO
3:00 AM	ECO	ECO	ECO	ECO	ECO	ECO	ECO
4:00 AM	ECO	ECO	ECO	ECO	ECO	ECO	ECO
5:00 AM	ECO	ECO	ECO	ECO	ECO	ECO	ECO
6:00 AM	ECO	IDLE	IDLE	IDLE	IDLE	IDLE	ECO
7:00 AM	ECO	IDLE	IDLE	IDLE	IDLE	IDLE	ECO
8:00 AM	ECO	IDLE	IDLE	IDLE	IDLE	IDLE	ECO
9:00 AM	ECO	IDLE	IDLE	IDLE	IDLE	IDLE	ECO
10:00 AM	ECO	IDLE	IDLE	IDLE	IDLE	IDLE	ECO
11:00 AM	ECO	IDLE	IDLE	IDLE	IDLE	IDLE	ECO
12:00 PM	ECO	IDLE	IDLE	IDLE	IDLE	IDLE	ECO
1:00 PM	ECO	IDLE	IDLE	IDLE	IDLE	IDLE	ECO
2:00 PM	ECO	IDLE	IDLE	IDLE	IDLE	IDLE	ECO
3:00 PM	ECO	IDLE	IDLE	IDLE	IDLE	IDLE	ECO
4:00 PM	ECO	IDLE	IDLE	IDLE	IDLE	IDLE	ECO
5:00 PM	ECO	IDLE	IDLE	IDLE	IDLE	IDLE	ECO
6:00 PM	ECO	ECO	ECO	ECO	ECO	ECO	ECO
7:00 PM	ECO	ECO	ECO	ECO	ECO	ECO	ECO
8:00 PM	ECO	ECO	ECO	ECO	ECO	ECO	ECO
9:00 PM	ECO	ECO	ECO	ECO	ECO	ECO	ECO
10:00 PM	ECO	ECO	ECO	ECO	ECO	ECO	ECO
11:00 PM	ECO	ECO	ECO	ECO	ECO	ECO	ECO

To achieve accurate expansion results, it is often necessary to rely on thermostatic control using a water cycle. This method requires continuous operation of the thermostat, which consumes a significant amount of energy and produces waste heat.

NETZSCH has succeeded in eliminating the external thermostat. The temperature of the measuring chamber is controlled electronically while maintaining excellent temperature stability. This significantly reduces the energy consumption of a DIL 502 *Expedis*®.*

Additionally, the user-defined scheduling function allows the system to transition between an Idle and Eco Mode as needed. This ensures that the gas flow is reactivated in time for the next scheduled measurement – without the need for a full restart and lengthy warm-up period.

* Reduction by up to 60%



Unlimited Warranty

At NETZSCH, our commitment to quality extends well beyond the instruments themselves. We understand that your investment in advanced technology is a long-term one, and that's why we offer something truly unparalleled – our Unlimited Warranty.

What Does Unlimited Warranty Mean?

Unlike other warranties that may have hidden limitations, NETZSCH's Unlimited Warranty proves our dedication to your success. For as long as it is technically possible, we stand by our instruments and support you with:

- **Attractive Contract Pricing:** Take advantage of the extraordinary price/performance ratio of our NETZSCH Unlimited Warranty.
- **Comprehensive Coverage:** From day one and throughout the lifetime of your instrument.
- **Expert Service:** Receive high-quality service directly from NETZSCH or our authorized dealers.
- **Predictable Costs:** With our maintenance contracts, you can plan your expenditures more reliably.
- **Long-Term Reliability:** Our Unlimited Warranty ensures that your instruments maintain their value and performance.

You can rely on our unparalleled support for your thermal analysis, rheology and fire testing needs.

https://netzs.ch/unlimited-warranty

Heating and Cooling Accessories

Furnace Type	Max. Temp.	Cooling System	Min. Temp.
Copper*	500°C	Vortex AIC 80 LN_2	0°C -60°C -180°C
Stainless Steel*	1000°C	Fan (included) AIC 80 Vortex LN_2	RT -40°C 0°C -150°C
Fused Silica	1150°C	-	RT
Silicon Carbide	1600°C	Fan (included)	RT
Silicon Carbide (fast cooling)	1600°C	Fan (included) Compressed air	RT
Rhodium*	1600°C	Fan (included)	RT
Graphite*	2000°C	Tap water*** Cooling thermostat	RT
Graphite**	2400°C	Tap water*** Cooling thermostat	RT
Graphite**	2800°C	Tap water*** Cooling thermostat	RT

* *Select* and *Supreme*
** *Supreme*
*** Requires connection to tap water

Furnaces are designed to accommodate a variety of atmospheres – whether inert, oxidizing (for graphite only up to 1680°C by using a special protective tube), reducing, or vacuum – thus providing optimal performance tailored to your product variations and temperature requirements.

A Range of Furnaces to Match Your Application

The DIL 502 *Expedis®* can be tailored to any application with its wide range of interchangeable furnaces. The silicon carbide furnace provides access to temperatures up to 1600°C for all DIL 502 *Expedis®* versions, making them suitable for routine measurements. For subambient requirements, the *Select* and *Supreme* versions offer coolable furnaces with different cooling options.

For high-temperature applications, three graphite furnaces reaching 2000°C, 2400°C and 2800°C are the appropriate configuration for measuring the thermal expansion of metals, alloys, ceramics or composites. This is ideal for industries such as aerospace, power generation, and oil and gas, as well as demanding research environments.

Dual Furnace Systems for Enhanced Productivity or More Flexibility

All DIL 502 *Expedis®* versions can be equipped with one or two furnaces. This provides maximum versatility. Choose identical furnaces for double the throughput, or combine different furnace types to cover a broader range of applications. The furnaces are interchangeable, and switching between them is quick and easy.

Master Every Material with Cooling Flexibility

The DIL 502 *Expedis®* range of dilatometers offers a variety of cooling options to precisely match your testing needs. This helps tailor the cooling process to mimic real-world conditions or specific manufacturing processes, ensuring the most relevant and accurate data.

Advanced cooling systems, like compressed air cooling, dramatically reduce the turnaround time between runs, maximizing productivity. Superior temperature uniformity and response time are crucial for such processes as rate-controlled sintering and the characterization of materials undergoing rapid temperature changes. Temperatures as low as -180°C/-150°C can be achieved using liquid nitrogen (LN_2). For cooling down to -60°C without liquid nitrogen, an air intracooling system, AIC, can be connected.

AIC 80 air Intracooler system

Controlled Environments

All DIL 502 *Expedis®* instruments are at least gas-tight. A controlled environment in dilatometric measurements:

- **Prevents unwanted reactions** using inert gases.
- **Simulates real-world oxidation;** creates specific reducing conditions.
- **Achieves pristine, oxygen-free environments with vacuum.**
  Consistent conditions also minimize variation, ensuring reproducibility and accuracy of measurements. This also protects the instrument from damage by preventing corrosion and contamination.

The DIL 502 *Expedis*® *Select* and *Supreme*

MEASURE UP TO 2000°C AND BEYOND

Pyrometer for Detection of the Highest Temperatures

W-Rh thermocouples react with graphite at very high temperatures. Therefore, the sample temperature of the DIL 502 *Expedis*® *Supreme* is measured optically with a high-performance pyrometer from room temperature onward up to 2400°C/2800°C.

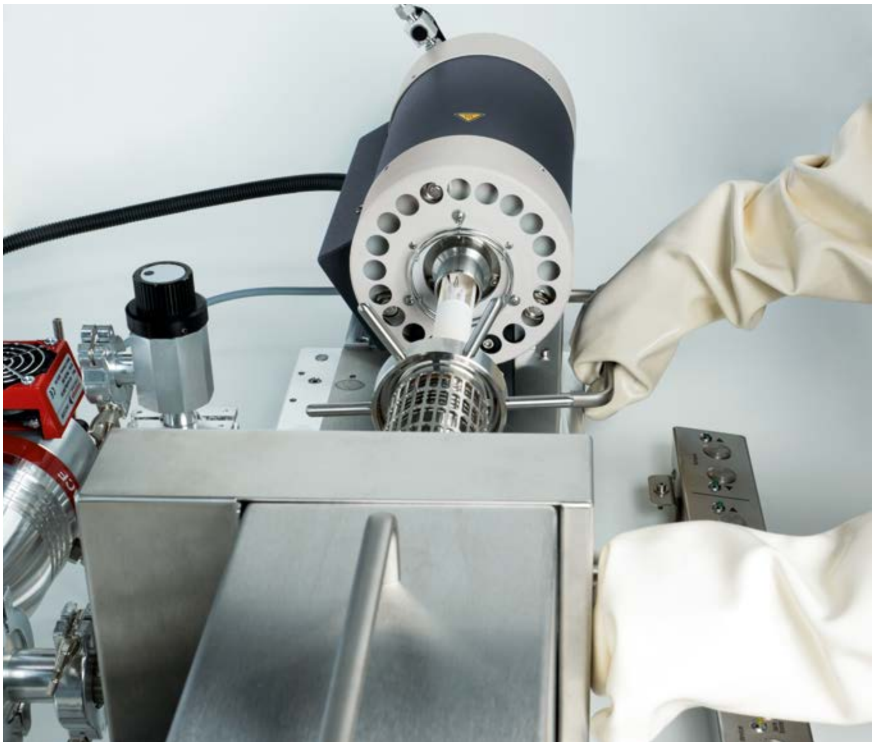
Refined Safety System

An elaborate safety system monitors the cooling water and purge gas flow throughout the measurement.

Variable Gas Atmosphere Is Key

With the graphite furnaces, the specimen chamber and furnace chamber are always separated by means of a protective tube (glassy carbon or alumina). This allows for the use of a different atmosphere around the specimen than around the heating elements. In combination with an alumina protective tube and an alumina sample holder, even an air atmosphere can be applied to the specimen up to 1680°C in the furnace.

The DIL 502 Expedis® Supreme reaches temperatures up to 2800°C



Inside the glovebox – DIL *Expedis® Supreme*

Comfortable Handling

The glovebox version of the DIL *Expedis® Supreme* enhances operator mobility with large, accessible buttons for smooth operation.

The system's electronics are positioned outside the glovebox, and an optional remote control unit allows for easy movement of the pushrod or furnace from outside, ideal when the dilatometer and electronics are out of reach.

DIL *Expedis® Supreme* Glovebox Version

For Investigations Requiring Special Care

In cases where materials are very sensitive to oxygen or humidity, or operators have to be protected from specimen properties, the challenge can often only be managed by using a glovebox. The DIL *Expedis® Supreme* glovebox version was specifically developed for such applications.

The entire casing of the dilatometer is made of stainless steel. There are no plastic parts that could potentially interact with specimens or the environment.

For measurements up to 1650°C (furnace temperature) in an argon atmosphere, NETZSCH offers a specially dedicated rhodium furnace.

DIL *Expedis® Supreme* glovebox version with separate control panel (option) for use inside the glovebox

Specimen Holders and Accessories

The DIL 502 *Expedis®* lets you choose from a variety of sample holders and accessories made of different materials. This maximizes the performance of your dilatometer and ultimately yields more reliable data.

Tube sample holder kit made of alumina for dual measuring system

Tube sample holder kit made of fused silica for single measuring system

Tube sample holder kit made of graphite for single measuring system

Tube sample holder kit made of alumina for single measuring system

Tension sample holder kit made of alumina

Higher Throughput and Efficiency – Dual Sample Holders

A dual sample holder allows simultaneous measurements of two specimens. This not only increases throughput, but also enables comparative studies of different materials under identical conditions. In differential mode, both the sample and the correction measurements can be conducted simultaneously. For the operation of a dual sample holder, a dual measuring system is needed.

The Best-Suited Sample Holder for Your Application

Each material used for sample holders has different thermal conductivity and expansion characteristics. For example, a graphite holder can withstand high temperatures and rapid cooling rates, making it ideal for high-performance applications, e.g., aerospace or energy sector. By selecting the appropriate holder material, you can achieve higher measurement accuracy and reproducibility, which is essential for research and development.

Sample Holder for Films, Foils and Fibers

Maintaining a stable position of the specimen in the sample holder is crucial for successful measurement results. For films, foils, and fibers, this can be achieved by using a tension sample holder.

Sample containers made of alumina, sapphire, fused silica and graphite

Sample container for measurements on wax

Protective sleeve made of molybdenum, aluminum nitride (AlN) and graphite

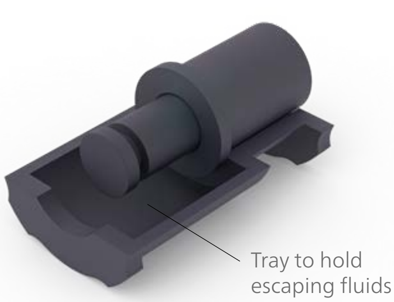
From Protective Sleeves to Special Containers for Critical and Oxygen-Sensitive Samples

The use of sample containers and protective sleeves is essential for preserving the integrity of both the specimens and the equipment.

The sample containers are designed for the measurement of samples in powder or paste form, or for the measurement of materials in the transition from a solid to a liquid state. Molten salts can be measured using a special graphite container. To avoid damage to the sample holder, the container support is equipped with a tray that collects leaking liquids.

When interactions between the specimen and the sample holder material are expected, protective sleeves can be used to prevent contact and contamination. These protective sleeves are made from different materials to address various needs and temperature ranges.

Sample container for molten salts



OTS® (Oxygen Trap System)

Tungsten specimen in sample holder plus Oxygen Trap System to prevent corrosion. This provides an oxygen-free atmosphere during the measurement.

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 is fast and efficient. Offering a large range of functions with a clearly-arranged user interface, it is intuitive and thus easy to learn.

This software has additional options, though, which impress even the most experienced operators – particularly its *Density Determination*, patented c-*DTA*®, Rate-Controlled Sintering and innovative *Identify* software features.

Density Determination

This software option allows for determination of the density and volumetric change of specimens based on the measured thermal expansion. It can be applied for solids, liquids or transitions from solid to liquid, among others.

Softening Point Detection

Upon reaching its softening point under a specified load and controlled heating rate, the material undergoes notable deformation, which is recorded as a shrinkage in the thermal expansion curve. After a defined value of shrinkage is recorded, the measurement is stopped to protect pushrod and specimen holder from damage and the pushrod automatically retracts away from the sample. This feature is important when measuring materials such as glass and glazes, which tend to soften when heated.

Patented* c-*DTA*®

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

* DE102013100686

Rate-Controlled Sintering (RCS)

RCS allows for control of the sintering rate during a DIL measurement by delivering precise, non-linear heating profiles that optimize your materials' microstructure and densification. The temperature program of the furnace is controlled so as to achieve the predefined sintering rate for the specimen. Depending on the selected RCS mode, the furnace may no longer be heated at a constant rate: The heating process may be stopped/started or continuously adapted depending on the sintering behavior of the specimen. The measured temperature profile can then be used for optimizing the production process.

Kinetics Neo – Process Optimization by Prediction

Rising energy costs and the demand for high-quality ceramics make process optimization essential. Kinetics Neo offers a precise, efficient solution that streamlines ceramic manufacturing, saving time and resources without sacrificing quality. The Kinetics Neo software offers a cutting-edge solution that combines precision and efficiency to revolutionize ceramic manufacturing, saving both time and resources without compromising quality.

Proteus® *Search Engine*

- Efficient data management
- Directly access and sort data by criteria
- Quickly view measurement and analysis previews without opening files
- Retrieve data quickly and easily
- Search by such criteria as instrument name, method, operator, file and signal type, date, measurement conditions or evaluated effects

Software Features

	Classic	*Select*	*Supreme*
Report Generator	■	■	■
Softening point detection	■	■	■
Automatic specimen length detection	■	■	■
Software-controlled force adjustment (incl. constant forces, ramps, steps)	■	■	■
Eco Mode	■	■	■
AutoEvaluation	■	■	■
Identify	□	□	■
RCS (Rate-Controlled Sintering)	□	□	■
c-*DTA*® (caloric effects or calibration)	□	□	■
Density Determination	□	□	■
Force modulation	□	□	■
Temperature modulation	□	□	□
Proteus® Search Engine	□	□	□
LabV®	□	□	□
Kinetics Neo	□	□	□

■ Included
□ Optional

LabV®*

- **Digital Workflows**
  Streamline your testing process with automated workflows and a highly intuitive interface.
- **Data Platform**
  Connect all your testing devices and IT systems for complete end-to-end process integration.
- **AI-Powered Digital Assistant**
  The first data platform to offer laboratories access to AI by using natural language

* LabV® Intelligent Solutions GmbH is a member of the NETZSCH Group

AutoEvaluation

Built-In Thermal Analysis Expert

AutoEvaluation

AutoEvaluation is an intelligent software functionality exclusively offered by NETZSCH. It is a self-acting evaluation of thermo-analytical measurement curves that works without using pre-defined macros. This is an immense support and time saver.

AutoEvaluation offers special functions for the evaluation of various materials. When testing metals, "Metal Melting" will automatically evaluate the onset of melting. "Glass Transition and Softening" displays the onset of glass transition and the peak of softening with just one click. In measuring ceramics, sintering steps will be displayed when the sintering of a specimen is detected.

AutoEvaluation is included when you purchase any instrument in the DIL 502 *Expedis*® range.

This intelligent feature operates independently of the operator's input, ensuring objective evaluation while simultaneously saving time.

Input Assistant for a Fast Start and Method-Based Automatic Evaluation

The *Proteus*® software allows for properties and methods from previously executed measurement files to be applied with a simple mouse click. The evaluation steps for a reference test run can be saved in a method and applied, fully automatically, to a specimen measurement after its termination. It is also possible to have the software highlight any results deviating from the selected quality criteria.

Identify

The *Identify* software extension helps identify and interpret dilatometer measurements, offering NETZSCH libraries with hundreds of entries from various fields. User-specific libraries can be shared via a network. *Identify* allows for the identification of unknown specimens from measured curves, using absolute values or shapes. It also enables comparisons to known specimens to determine principal material behavior, with all measurements stored for future identification or quality comparison.

Quality Control

Via agreement between the current measurement and selected database entries.

Database Functionality

Archiving of current measurements and the search for suitable measurement conditions for future tests from existing entries in the database.

Measurement Update in Passing – LED Status Bar

The DIL 502 *Expedis*® series provides an LED light bar that allows you to check the status of your instrument as you walk by, with different colors representing different statuses. It is reassuring to see from afar, without having to log into your PC, that your measurement is running smoothly and to be able to read instrument status notifications such as:

- Instrument is ready
- Measurement is running
- Measurement progress
- Heating/cooling to setpoint
- User interaction needed
- A problem occurred

Improving Your Productivity and Workflow with Innovative User Interface

The color touch lets you start a measurement using the NETZSCH *Proteus*® software with the touch of a finger, offering the ability to also:

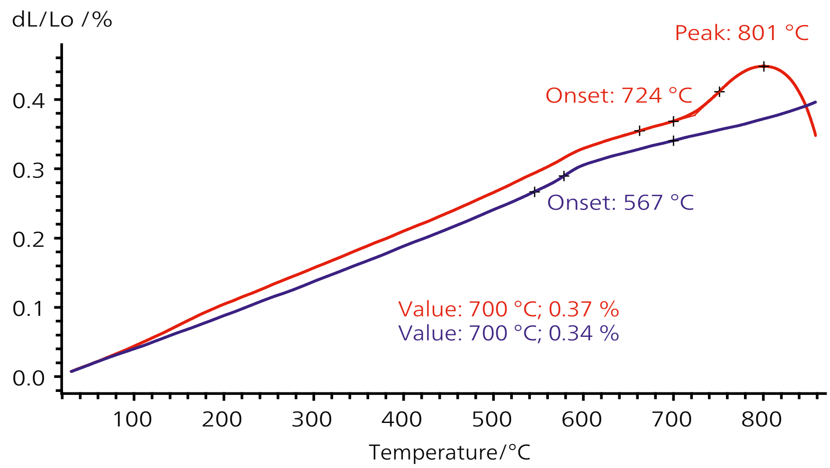
- See the progress of your measurement and time remaining
- Check recently finished measurements
- Check and change gases and set point state
- Get an immediate overview of the evaluated measurement
- Configure and start *AutoVac*, and check its status
- Change static force
- Detect sample length
- Select sample holder

Display showing *AutoEvaluation* after measurement has finished.

Applications

Thermal Expansion Mismatch

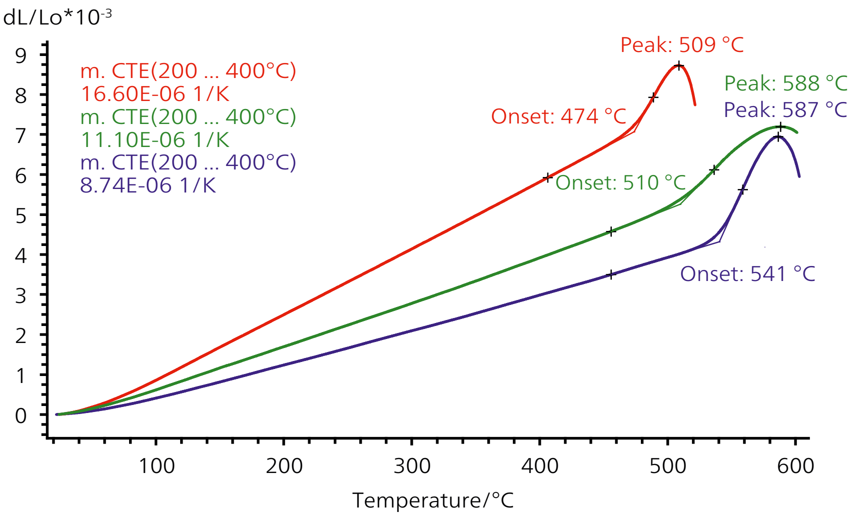
Poor glaze/body fit is the main cause of crazing (spider web pattern of cracks penetrating the glaze). This effect can result from such cases as thermal expansion mismatch, which can be avoided by adjusting the thermal expansion behavior of the body and the glaze. This plot shows the expansion behavior of the glaze (red curve) compared to that of the body to which it should be applied. At 700°C – shortly before reaching the glass transition temperature of the glaze at 724°C – the difference in expansion amounts to 0.03%. Softening of the glaze occurs at 801°C. Crazing occurs when the glaze is under tension (stretched) during cooling. This can happen when the glaze expands more than the body.



Comparison of the thermal expansion of a glaze and the body on which it should be held. α→β transition of quartz is detected in the curve of the body at 567°C (extrapolated onset). Measurement conditions: Heating rate 5 K/min, air atmosphere.

The ideal scenario is when the glaze and body expand and contract at the same rate. This is rarely achieved in practice. This is when dilatometry comes into play.

CLASSIC



Thermal expansion curves of three different glass samples.
Measurement conditions: Heating rate 5 K/min, air atmosphere.

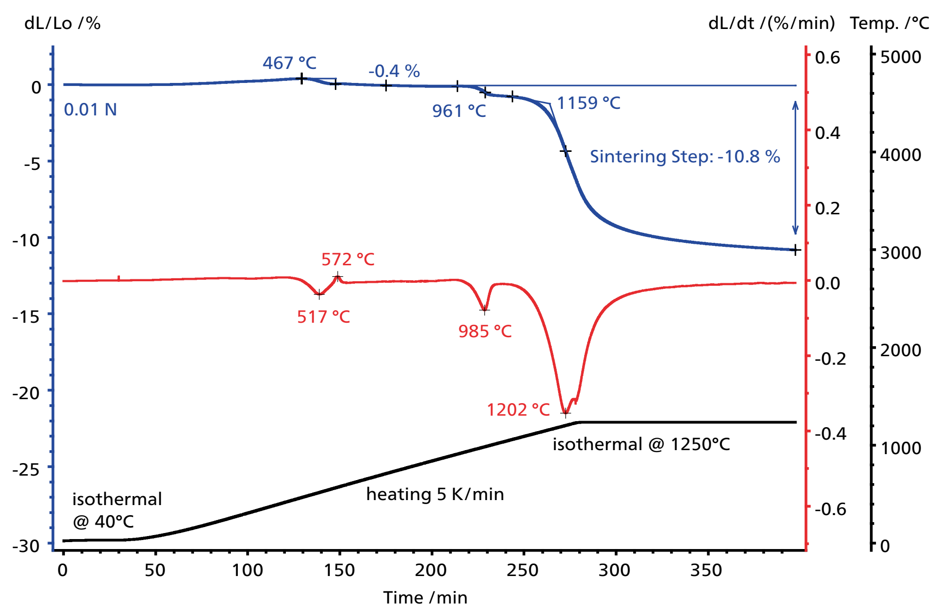
Determination of Quality Parameters in Glass Production

Thanks to their excellent optical, chemical and mechanical properties, glasses can be used to make high-quality products such as implantable medical devices and devices used in space exploration. This plot illustrates the thermal expansion of three different types of glass between room temperature and 600°C, highlighting their distinct expansion and softening behaviors. In such instances, dilatometry serves as an ideal tool for studying the influence of varying compositions and production conditions on thermal expansion.

Understanding Sintering Behavior

Porcelain is a ceramic made mainly of kaolinite, feldspar, and quartz. When fired above 1200°C, it forms glass and mullite, giving it 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 (at 467°C in the blue thermal expansion curve, related to the peak at 517°C in the red 1st derivative curve). This temperature range marks the release of chemically bound water from the clay structure, causing about 0.4% shrinkage. 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 be 10%.

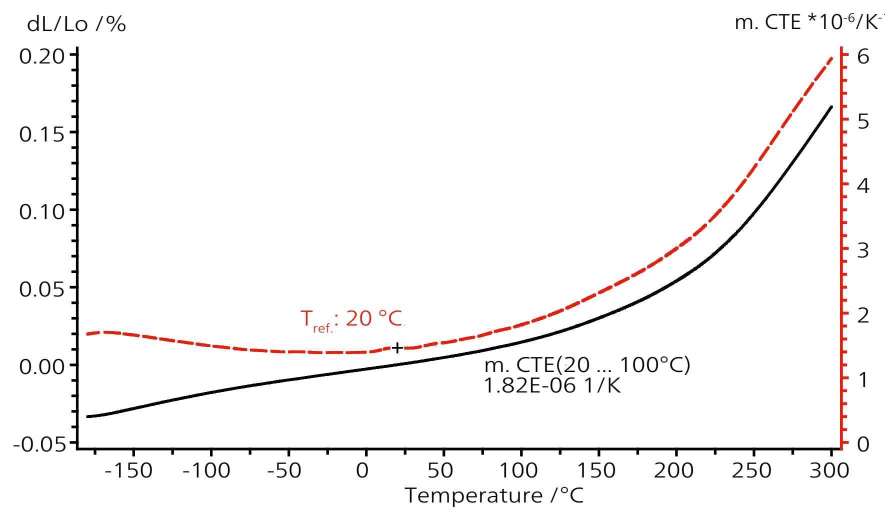


Porcelain green body. Measurement conditions: RT to 1250°C, heating rate 5 K/min, dynamic air atmosphere (20 ml/min), contact force of the pushrod 0.01 N

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

Measurement of the Smallest Thermal Expansions on an Invar Alloy

Invar 36® is a nickel-iron alloy with 36% nickel, known for its exceptionally low thermal expansion – about one-tenth that of carbon steel. It maintains dimensional stability from cryogenic temperatures up to 227°C, offering toughness, versatility, and reliable strength at low temperatures. Consequently, it is used in applications where high dimensional accuracy is required despite temperature fluctuations. The figure on the right shows the thermal expansion and coefficient of thermal expansion (CTE) as a function of temperature. During heating, a CTE of 1.82 × 10^{-6} 1/K was measured in the temperature range from 20°C to 100°C.

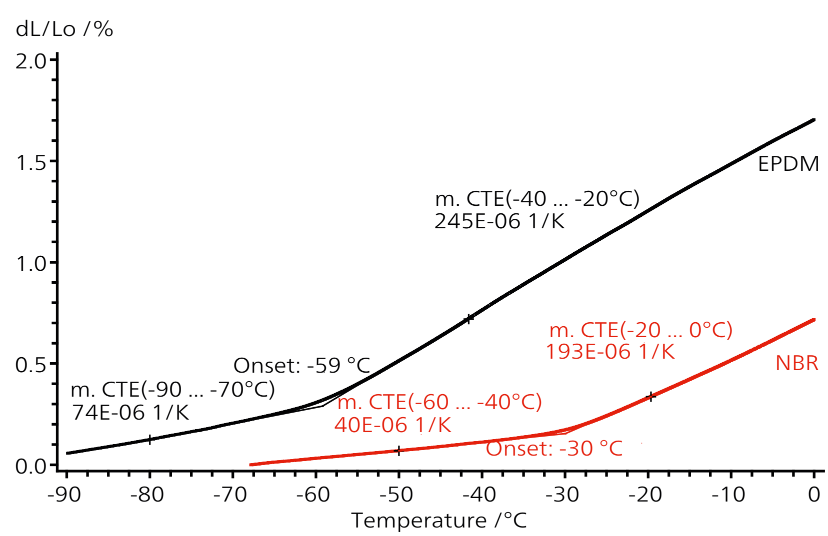


Thermal expansion (solid line) and mean CTE (dashed line) between -180°C and 300°C of Invar 36®; heating rate: 5 K/min, He atmosphere, fused silica sample holder

SELECT

Determination of the Thermal Expansion of Elastomers

Precise knowledge of the glass transition temperature and thermal expansion is required when testing technical elastomers. This example shows the linear thermal expansion of ethylene propylene diene monomer (EPDM) and acrylonitrile butadiene rubber (NBR). Depending on the cooling option connected to the DIL 502, different temperature ranges below room temperature can be recorded. In this example, EPDM was measured using an LN_2 cooling option (down to at least -180°C), while NBR was measured using a two-stage air cooler (AIC 80; down to at least -60°C).



The thermal expansion and mCTE values of EPDM and NBR; heating rate: 1 K/min, He atmosphere, fused silica sample holder

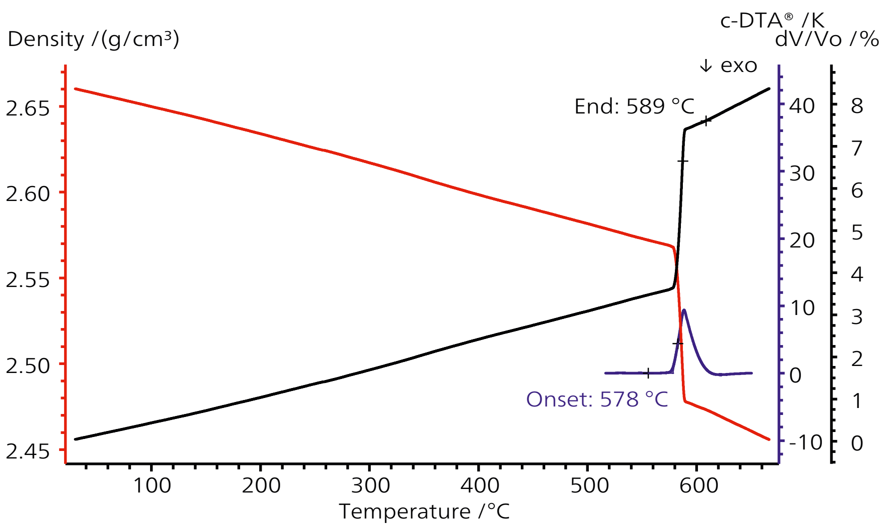
Volumetric Expansion of an Aluminum Alloy into the Melt

The behavior of an aluminum-based alloy during heating is illustrated here. Displayed are the volumetric expansion (dV/V_0, black) and the density change (red) which can both be calculated from the measured thermal expansion data by using the NETZSCH *Density Determination* software.

Following an initial linear expansion, the aluminium alloy begins to melt at 578°C (the extrapolated onset temperature of the c-*DTA*® signal, shown in blue). Such an experiment requires a special container (here, alumina). During melting, strong expansion occurs, represented by the mushy region in which the liquid and solid states are present together. Above 589°C, the entire sample is completely melted.

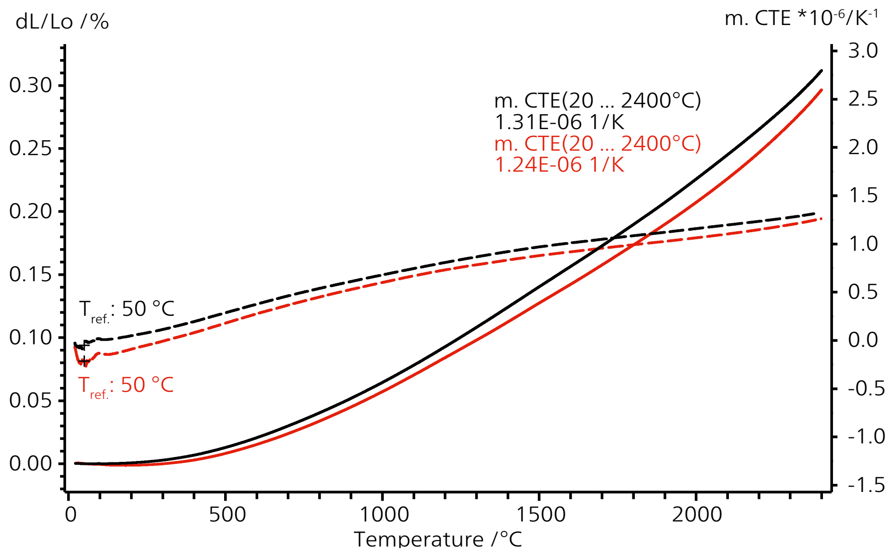
As the volume increases, the initial density decreases by around 9% (from 2.66 g/cm³ to 2.46 g/cm³) until the end of the measurement.

The c-*DTA*® curve (blue) clearly shows the melting range via endothermal effects.



Thermal behavior of an aluminum-based alloy. Measurement conditions: heating rate: 5 K/min, He atmosphere, constant contact force: 250 mN, alumina sample holder, alumina container. Displayed are the volumetric expansion (black line), the curve of the calculated density change (red line), and the c-*DTA*® curve (blue line).

Thermal Behavior of Carbon Fiber-Reinforced Carbon up to 2400°C



Thermal expansion (solid lines) and mean CTE (dashed lines) between 20°C and 2400°C of two CFC samples measured 45° (black) and 0° (red) relative to the fiber direction; heating rate: 5 K/min, He atmosphere, graphite sample holder

This composite material consists of a pure carbon matrix, reinforced with carbon fibers. It exhibits high mechanical strength and high-temperature stability. The thermal expansion of C/C materials depends on their fiber architecture.

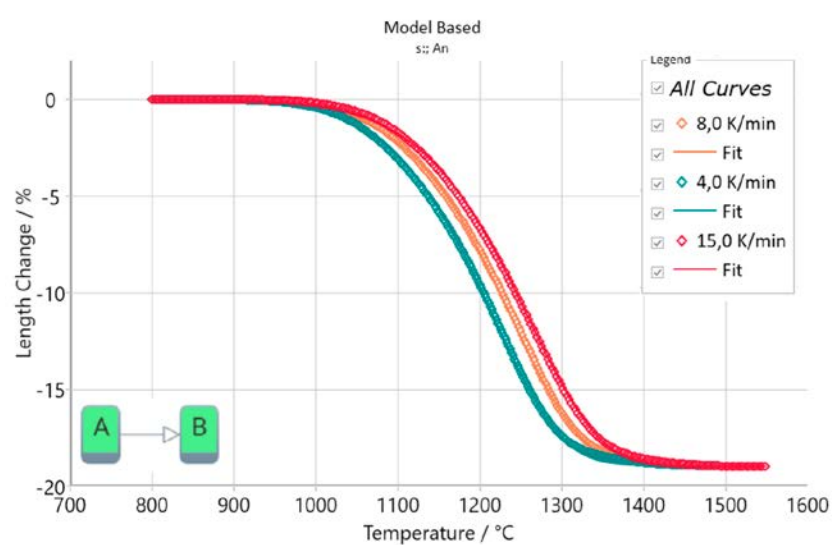
In this case, experiments were conducted at 45° (black) and 0° (red) angles relative to the fiber orientation. The mean CTE of the specimen, cut at a 45° angle relative to the fiber direction (black) between 20°C and 2400°C, is just 0.07 E-06 K^{-1} higher than that of the sample cut at a 0° angle. This suggests that the material properties depend very little on these spatial directions.

SUPREME

Optimization of Sintering Processes

Optimized and Superior Quality – Redefining Zirconia Green Bodies for Dental Solutions

The firing of ceramics is accompanied by a length decrease, where the sintering rate depends on the heating rate. The figure presents dilatometer measurements (symbols) at different heating rates of 4, 8 and 15 K/min after subtracted linear thermal expansion and arriving at a final length decrease of 18.9%. By using Kinetics Neo software, the common kinetic model of the Avrami nucleation type is created for these experimental curves. For each experimental heating rate, the length change is simulated by the kinetic model (solid curves in the figure) and has very good agreement with experimental length curves.
This kinetic model can also be used for sintering predictions for other temperature profiles, as well as for the optimization of temperature profiles in industrial processes.

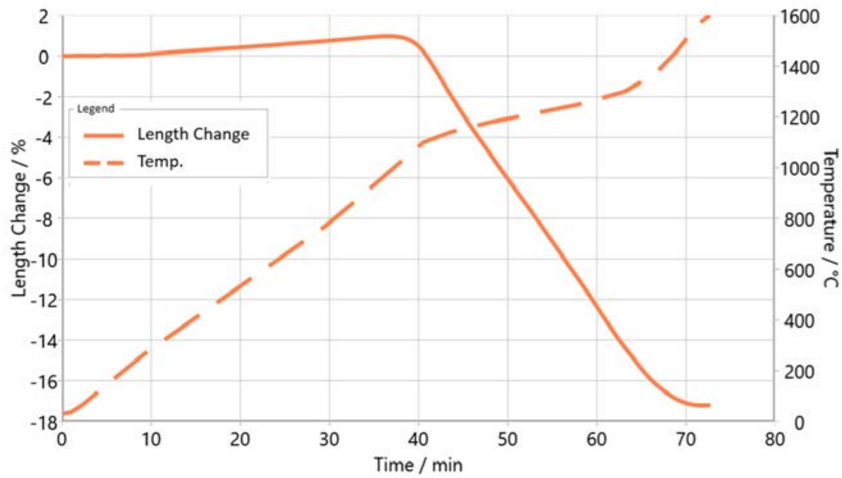


Dilatometer measurements (rhombus symbols) and kinetic model (solid lines) of sintering for zirconia green body at heating rates of 4, 8, and 15 K/min.

KINETICS NEO

By combining dilatometer measurements with advanced kinetic modeling using Kinetics Neo software, the sintering process can be optimized to significantly improve product quality, enhance cost efficiency, and accelerate development and scale-up.

The sintering process can be accurately modelled in Kinetics Neo software using various temperature profiles, or by controlling constant shrinkage rates, eliminating the need for lengthy and costly trial-and-error experimentation. The figure illustrates the optimized temperature (dashed line) tailored to enhance the sintering performance of ceramic materials with a constant rate of sintering degree of 3.5%. The solid line is the corresponding measured length change for this profile. As expected from simulation, the measured shrinkage rate is constant. The optimized sintering time is reduced by more than 50%.



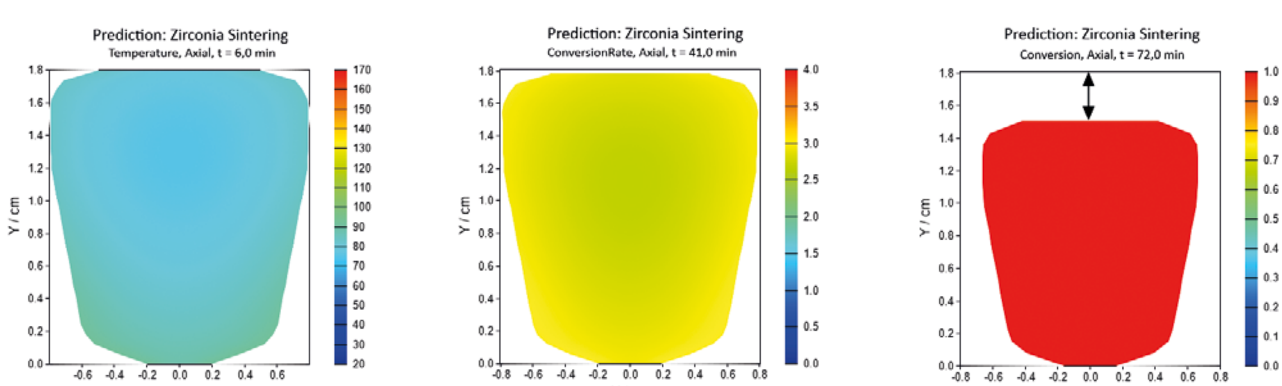
Optimized temperature profile for ceramic sintering (dashed) and measured length change (solid) for verification of this profile

TERMICA NEO

For complex geometric components, Termica Neo simulates thermal shrinkage and temperature gradients.

Using the Termica Neo software, the simulation for sintering inside the material can be carried out including temperature gradients, conversion and sintering rate in each point of sintered volume. Here, the optimized temperature profile is selected as the surrounding temperature. (A) illustrates the temperature at t = 6.0. The sintering rate at time = 41.0 min (B) is higher and slightly depends on coordinates. (C) presents the degree of conversion after a firing cycle of 71.0 minutes, where the red color and the decreased linear size signify complete sintering.

(A) (B) (C)



Simulation of dental ceramic for optimized temperature profile. Vertical cross-sections for temperature at t = 6.0 min (A), conversion rate at t = 41.0 min (B), and degree of conversion at t = 72.0 min (C)

DIL 502 *Expedis*®			
	Classic	*Select*	*Supreme*
Design	▪ Horizontal, pushrod ▪ Single or dual furnace operation	▪ Horizontal, pushrod ▪ Single or dual furnace operation	▪ Horizontal, pushrod ▪ Single or dual furnace operation***
Instrument interface	Illuminated information panel (optional touch display)	Touch display	Touch display
Temperature range	RT … 1600°C	-180°C … 2000°C	-180°C … 2800°C
Heating rates	0.001 … 50 K/min	Depending on furnace type: 0.001 … 50 K/min Graphite: 0.001 … 100 K/min	
Cooling systems	Depending on furnace: Air compressor	Depending on furnace: Vortex, LN_2 device, air compressor	Depending on furnace: Vortex, LN_2 device, air compressor
User-interchangeable specimen holder systems	SiO_2, Al_2O_3, graphite, user-interchangeable ▪ Single system (one pushrod) ▪ Dual system with two pushrods usable in dual or differential mode	SiO_2, Al_2O_3, graphite, user-interchangeable ▪ Single system (one pushrod) ▪ Dual system with two pushrods usable in dual or differential mode	SiO_2, Al_2O_3, graphite, user-interchangeable ▪ Single system (one pushrod) ▪ Dual system with two pushrods usable in dual or differential mode
Specimen dimensions	Length: Max. 52 mm Ø 12 mm standard (optional Ø 19 mm max.) Ø 8 mm in dual specimen holder system	Length: Max. 52 mm (Graphite furnace: 25 mm) Ø 12 mm standard (optional Ø 19 mm max.) Ø 8 mm in dual specimen holder system	Length: Max. 52 mm (Graphite furnace: 25 mm) Ø 12 mm standard (optional Ø 19 mm max.) Ø 8 mm in dual specimen holder system
Gas atmosphere	Inert, oxidizing under static or dynamic conditions	Inert, oxidizing**, reducing, vacuum	Inert, oxidizing**, reducing, vacuum
Gas control	3-way switch or 1-/3-/4-way MFC*	1-way MFC or 3-/4-way MFC*	1-way MFC or 3-/4-way MFC*
Gas-tight	Yes	Vacuum-tight	Vacuum-tight
Oxygen Trap System (*OTS*®)	No	Optional, for single and for dual specimen holder systems	Optional, for single and for dual specimen holder systems

DIL 502 *Expedis*®			
	Classic	*Select*	*Supreme*
Temperature accuracy	1 K	1 K	1 K
Temperature precision	0.1 K	0.1 K	0.1 K
Temperature resolution	0.001 K	0.001 K	0.001 K
Thermal stability (isothermal)	± 0.02 K	± 0.02 K	± 0.02 K
Measuring range	± 5000 µm	± 10000 µm	± 25000 µm
ΔL Resolution	2 nm	1 nm	0.1 nm
$\Delta L/L_0$ Repeatability	0.002%, absolute value	0.001%, absolute value	0.001%, absolute value
$\Delta L/L_0$ Accuracy	0.003%, absolute value	0.002%, absolute value	0.002%, absolute value
Force range (load at the specimen)	10 mN … 3 N (compressive and tensile force, depending on sample holder)		
Force resolution	0.001 mN	0.001 mN	0.001 mN

* optional
** graphite furnace: measurements in oxidizing atmosphere possible up to 1680°C by using a special protective tube
*** for the 2400°C and 2800°C graphite furnaces, only single furnace operation is possible

Technical Specifications

The owner-managed NETZSCH Group is a leading global technology company specializing in mechanical, plant and instrument engineering.

Under the management of Erich NETZSCH B.V. & Co. Holding KG, the company consists of the three business units Analyzing & Testing, Grinding & Dispersing and Pumps & Systems, which are geared towards specific industries and products. A worldwide sales and service network has guaranteed customer proximity and competent service 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 60 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.

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