Dilatometer Series
DIL 402 Expedis® Select, Supreme and Supreme HT
Method, Instruments, Applications

Analyzing & Testing
Dilatometry
The Method for Determination of Dimensional Changes

Each time a material is exposed to temperature changes – it shows a variation in its dimension. Whether it is in the course of its regular thermal expansion, by passing a phase transition or while it undergoes sintering, the substance will either be shrunk or elongated.

Dilatometry is the method of choice to study length change phenomena of ceramics, glasses, metals, composites, and polymers as well as other construction materials, thus revealing information regarding their thermal behavior and about process parameters or sintering (and curing) kinetics.

For preparing a dilatometer measurement, the defined sample is inserted into a sample holder and brought into contact with the pushrod. After closing the furnace, the experiment can be started.

Thermal expansion of the sample during heating is detected by the displacement system which the pushrod is connected to.

### Results obtainable by 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
- Optimizing of firing process
- Caloric effects by using c-DTA®
- Rate-Controlled Sintering (RCS)
- Kinetics Neo
Dilatometry Redefined

**MAXIMUM FLEXIBILITY**

The double furnace sliding carrier of the DIL 402 Expedis® Select/Supreme standard versions creates the opportunity to cover the entire temperature range from -180°C to 2000°C or, alternatively, to increase the sample throughput by having two furnaces available for use.

**UTMOST VERSATILITY**

Due to the wide dynamic range of the measurement system, it is possible to measure both soft and hard samples without impairment of the properties. Additionally, it enables force modulation and builds a bridge to thermo-mechanical analysis (TMA).
NanoEye – LARGEST MEASURING RANGE, HIGHEST RESOLUTION

The new, pioneering opto-electronic NanoEye displacement system features perfect linearity and maximum resolution over a measuring range which was impossible to realize until now.

<table>
<thead>
<tr>
<th>Instrument Type</th>
<th>Resolution</th>
<th>Measuring Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>Select</td>
<td>1 nm</td>
<td>± 10 mm</td>
</tr>
<tr>
<td>Supreme</td>
<td>0.1 nm</td>
<td>± 25 mm</td>
</tr>
</tbody>
</table>

USER-OPTIMIZED DESIGN

A wide force range for more fragile samples, built-in mass flow controllers and electrical thermostatting of the measuring cell to suppress any temperature influence from the surrounding provide for ease-of-use and maximum operational safety.
In classical dilatometry, the two parameters measurement range and resolution mostly seem diametrically opposed. If the resolution goes up, the measuring range usually goes down and vice versa.

*NanoEye*, the novel opto-electronic displacement system, is able to overcome this conflict and offers highest resolution associated with an unmatched measuring range.

**Functional Principle**

During a test run, if the sample expands, all green components in the graphics move backwards with the help of a linear guide (marked in blue). The optical encoder determines the corresponding length change directly on the appropriate scale.

**The NanoEye consists of:**

- an actuator which applies a controlled contact force and moves the pushrod for adjusting variable sample lengths
- an elastic force sensor which detects the contact force subsequently enabling a force control cycle
- an optical encoder (plus scale) which measures the initial sample length and determines the length change of the sample
Advantages using *NanoEye*

**Perfect Linearity**
compared to conventional transducer systems – for measurements with large thermal expansion and unmatched linearity.

**Wider Measuring Range Than Ever Before**
up to a factor of 10 compared to traditional dilatometers – for measurements on a large variety of different sample lengths with different thermal expansion behavior without manual adaption of measuring range.

**Friction-Free Construction**
without sliding or rolling friction and stick-slip effects – for highest reproducibility of results.

**Displacement Determination with Nanometer Resolution**
over the entire measuring range – for detecting even the smallest effects at every temperature.

**Controlled Contact Force During the Entire Measurement**
for measurements on small, delicate, fragile or foamed samples without risk of non-reproducible deformation.

**Extremely Small Forces**
adjustable for measurements on green bodies or soft samples.

**Maintenance-Free**
Optimum Adaptability

One or two furnaces, manual or motorized furnace operation, single or dual sample holders, tube type or rod type sample holders … these are only some of the features the Expedis® Select or Supreme provide to match nearly all application scenarios.

High Sample Throughput

The combination between the double furnace design and a dual sample holder used in dual mode increases the number of possible measurements tremendously and boosts the instrument’s efficiency.

The two standard versions of the DIL 402 Expedis® are specially designed for both research & development and sophisticated industrial applications: The comprehensive, fully-equipped Supreme model and the upgradable Select type (for a detailed comparison between the two models see page 25).

Designed to Master the Challenges of the Future
Greatest Variability in Contact Force

The DIL 402 Expedis® series is the first horizontal dilatometer series on the market which allows for force modulation and, by this means, bridges the gap between dilatometry and thermomechanical analysis under oscillatory load.

For test runs under static force conditions, different contact forces can be selected. Therefore, both models are ideally suited to measure not only soft samples but also rigid, fragile materials.

Widest Temperature Range from -180°C to 2000°C

To cover this temperature range, different furnaces are available and can be used both in the single furnace and double furnace format.

<table>
<thead>
<tr>
<th>Furnace Type/Heating Element</th>
<th>Max. Temperature Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>Copper</td>
<td>-180°C … 500°C</td>
</tr>
<tr>
<td>Stainless Steel</td>
<td>-150°C … 1000°C</td>
</tr>
<tr>
<td>Fused Silica (SiO₂)</td>
<td>RT … 1150°C</td>
</tr>
<tr>
<td>Silicon Carbide (SiC)</td>
<td>RT … 1600°C</td>
</tr>
<tr>
<td>Graphite</td>
<td>RT … 2000°C</td>
</tr>
</tbody>
</table>

Best Features of DIL 402 Expedis® Select/Supreme – Standard Versions
Smart Usability
Means More Than Just “Easy to Use”

Automatic Sample Length Detection

Measuring the sample length with a caliper runs the risk of result scatter, especially for soft samples. The DIL 402 Expedis® is capable of detecting the initial length of a sample automatically prior to the start of a test run under conditions identical to those during the measurement itself.

Large Measurement Range with Constant Resolution

In the past, it was often necessary to adapt the measuring range to the expected expansion or shrinkage of the sample to avoid signal overflow. With the new DIL 402 Expedis®, this is no longer the case. There is just one measuring range which is wide enough to quantify even the largest dimensional changes with a constant high resolution.

Perfect Temperature Stability of the Measuring System

Thanks to the elaborate electrical temperature control of the NanoEye, the measurement signal is not affected by environmental temperature fluctuations.
Temperature Measurement at the Right Spot

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

*MultiTouch*

A stable position of the sample inside the sample holder is a deciding factor for successful measurement results. The *MultiTouch* feature places the sample into the optimum position using a unique, tail-like motion.
Separate Gas Paths for Protective and Purge Gases

When using the three mass flow controllers (MFC, optional), the gas flow paths inside the instrument are split: the protective gas first passes through the measuring cell and then enters the sample chamber, whereas the purge gas(es) 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 MFC is integrated, the gas takes the same path as the protective gas mentioned above.

Schematic of the gas paths inside the instrument when using one protective gas and two purge gases
Oxygen-Free Measurement for Investigating Metals and Alloys

During the test run, in order to keep the residual oxygen concentration at the lowest possible level, the OTS® (Oxygen Trap System) can be applied. A getter ring on a ceramic substrate is mounted in the sample carrier tube and traps all oxygen residue within the inert purge gas.

Identifying by Evolved Gas Analysis

The vacuum-tight design of the DIL 402 Expedis® is ideally suited for connection to a QMS or to an FT-IR via capillary coupling to the SiC furnace. Outgassing of impurities, additives, organic binders and/or decomposition products can thus be studied.

Vacuum-tight Design for Best Sample Conditions

The instrument can be equipped with evacuation systems such as AutoVac for fast evacuating and gas-refilling as well as measurements under vacuum.
In cases where materials are very sensitive to oxygen or humidity, or operators have to be protected from sample properties, the challenge can often only be managed by using a glovebox. The DIL 402 Expedis® Supreme Glovebox Version was specifically developed for such applications. It is a must for gloveboxes which cannot be opened anymore after implementation.

The entire casing of the dilatometer is made of stainless steel. There are therefore no plastic parts to potentially interact with samples or the environment.

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

Working in a glovebox often significantly restricts an operator’s mobility, but the glovebox version of the DIL 402 Expedis® Supreme provides large, easily accessible buttons and allows for smooth operation.

The electronics of the system are separate from the mechanical parts wherever possible and designed for being positioned outside the glovebox.

For additional convenience, a remote control unit (optional) allows for controlling the movement of the pushrod or the movement of the furnace (optional) from outside the glovebox. This is especially advantageous if the dilatometer and the electronics are not together within the user’s operational radius.

Alternatively, a separate control panel for working inside the glovebox, with large buttons and stainless steel casing, is available. It can be positioned by the user wherever it fits and handled with gloves. There is therefore no need to remove the gloves in order to move the pushrod or the furnace.

The MultiTouch function (see page 11) ensures that the sample is in an optimum position after insertion, eliminating any need to tap on the sample holder or the instrument.

The sample holders are available as single or dual systems. In order to make sample holder exchange as simple as possible, the furnace is rotatable and only toggle screws or knurled screws, which can be handled easily with gloves, are used.
DIL 402 Expedis® Supreme HT
up to 2800°C

**Broadest Application Range**

Two graphite furnaces with end temperatures of 2400°C and 2800°C provide the appropriate configuration for measuring the thermal expansion of metals, alloys, ceramics and composites in applications such as aerospace, power generation, the oil and gas industry or demanding research. However, via an adapter, each of these graphite furnaces can be replaced with a standard furnace such as SiC, SiO₂, Cu or steel.

The opposite is also feasible: A DIL 402 Expedis® Supreme HT measuring part with a standard furnace can be subsequently retrofitted with a high-temperature furnace.

**Pyrometer for Detection of the Highest Temperatures**

Since W-Rh thermocouples can react with graphite above 2000°C, the sample temperature of the DIL 402 Expedis® Supreme HT is measured optically with a high-performance pyrometer from room temperature onward.

**Variable Gas Atmosphere Is Key**

In the DIL 402 Expedis® Supreme HT instrument, the sample 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 sample than around the heating elements. In combination with an alumina protective tube (maximum furnace temperature: 1680°C), even an air atmosphere can be applied to the sample.

**Refined Safety System**

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

The optional AutoVac system for evacuating and refilling the sample chamber along with integrated purging of the pyrometer window effectively supports the direct switch between oxidizing and inert conditions.
Recrystallized Silicon Carbide up to 2400°C

Recrystallized SiC (RSIC) sintered material is a technical ceramic often manufactured at temperatures around 2400°C. During sintering, a mixture of fine- and coarse-grained powder is transformed nearly shrinkage-free to a compact SiC matrix.

In the present case, the sample shows solely expansion over the entire temperature range from RT to 2400°C with CTE values of $4.337 \times 10^{-6}$ 1/K (between 30°C and 1000°C) and $4.960 \times 10^{-6}$ 1/K (between 30°C and 2000°C).
The unique Proteus® 7 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® and the innovative Identify software features. (More about Identify on pages 20/21).

**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
Special Features of the Proteus® Software for DIL 402 Expedis® Select and Supreme at a Glance*

Software-controlled force adjustment
(incl. constant forces, ramps, steps)

Force modulation

Density Determination

c-DTA® for temperature calibration or determination of caloric effects

RCS Rate-Controlled Sintering

Identify identification of unknown ΔL/L₀ curves through database comparison

Advanced Software
(for extended evaluation of the measuring data)*

Kinetics Neo

PeakSeparation (for processing the 1st derivative)

* for information which software features are included as standard and which ones are optionally available, please see page 25
Identify

Built-In Thermal Analysis Expertise

The unparalleled Identify, 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 absolute values, the slope or the shape of a measured curve. This will also open up the opportunity to compare known samples against a variety of other samples, enabling one to make a statement about material quality. Finally, all measurements can be stored in the extensive database and are always available for identification or quality comparison.
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
This composite material is composed of a matrix of pure carbon to which carbon fibers are added. It exhibits high mechanical strength associated with high temperature stability.

The thermal expansion of C/C materials depend on their fiber architecture. In the present case, experiments with angles of 45° (black) and 0° (red) relative to the fiber orientation were conducted. Both curves depict a characteristic behavior of such fiber-reinforced composites: passing a length change minimum between approx. 300°C and 400°C followed by expansion.

At 2000°C, the relative length change (dL/L₀ in %) as well as the corresponding mean CTE of the specimen cut out with a 45° angle relative to the fiber direction (black) are just about 7% higher (0.155% and 0.781 x 10⁻⁶ 1/K) than that of the sample cut out with a 0° angle (red, 0.144% and 0.727 x 10⁻⁶ 1/K). This suggests a quite low dependency of the material properties within these spatial directions.

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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.

After an initial linear expansion, the aluminum alloy starts to melt at 559°C (extrapolated onset temperature of the c-DTA® signal in dashed blue). For realizing such an experiment, a special container (here alumina, see photo) is necessary.

During melting, a strong expansion occurs representing the mushy region in which liquid and solid state are present together. Above 622°C, the entire sample is molten.

While the volume increases, the initial density drops down for about 10% (from 2.66 g/cm³ to 2.40 g/cm³) until the end of the measurement.

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

Thermal behavior of an aluminum-based alloy, heating rate: 5 K/min, He atmosphere, constant contact force: 250 mN, alumina sample holder, alumina container. Displayed are the volumetric expansion (black solid line), the curve of the calculated density change (red solid line) as well as the c-DTA® curve (blue dashed line).
Tungsten is a metal very sensitive to oxidation. But due to the vacuum-tight design of the Expedis® Supreme, the material can be measured in pure He atmosphere (in combination with OTS® – Oxygen Trap System) to get its true expansion behavior. There is no need of reducing atmosphere to suppress superficial oxidation (which would change the color of the sample).

**Comparison of Measurement and Literature**

| Measured CTE between 20°C and 1500°C | 5.143 x 10⁻⁶ 1/K |
| Literature values (NIST standard table) | 5.129 x 10⁻⁶ 1/K |
| Difference between measurement and literature | 1.4 x 10⁻⁸ 1/K |

Thermal behavior of tungsten, sample length: 25.00 mm, heating rate: 5 K/min, He atmosphere, constant contact force: 250 mN, alumina sample holder. Displayed are the length change of the sample (black solid line) together with the tabulated theoretical data (red dashed line, NIST standard table).

The picture on the left site illustrates two tungsten samples after the experiment. The used tungsten samples were measured up to 1640°C. The right sample is corroded because of non-oxygen free atmosphere during measurement. The left sample, also shown in the picture above, is still shiny due to a measurement in oxygen-free atmosphere.
## Configurations

<table>
<thead>
<tr>
<th>Feature</th>
<th>Supreme</th>
<th>Select</th>
<th>Supreme HT</th>
</tr>
</thead>
<tbody>
<tr>
<td>Max. temperature range</td>
<td>-180°C … 2000°C</td>
<td>-180°C … 1600°C</td>
<td>(-180°C)* … RT … 2800°C</td>
</tr>
<tr>
<td>Measuring range</td>
<td>50 mm (± 25 000 µm)</td>
<td>20 mm (± 10 000 µm)</td>
<td>50 mm (± 25 000 µm)</td>
</tr>
<tr>
<td>ΔL Resolution (over entire measuring range)</td>
<td>0.1 nm</td>
<td>1 nm</td>
<td>0.1 nm</td>
</tr>
<tr>
<td>Double furnace sliding carrier</td>
<td>✓</td>
<td>✓</td>
<td>N/A</td>
</tr>
<tr>
<td>Motorized furnace operation</td>
<td>□</td>
<td>□</td>
<td>□</td>
</tr>
<tr>
<td>Vacuum-tight design</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
</tr>
<tr>
<td>Automatic Evacuation System – AutoVac</td>
<td>□</td>
<td>□</td>
<td>□</td>
</tr>
<tr>
<td>Mass Flow Controller (MFC) – single/triple</td>
<td>✓ / □</td>
<td>✓ / □</td>
<td>✓ / □</td>
</tr>
<tr>
<td>Available Cooling Devices</td>
<td>Vortex, LN₂</td>
<td>Vortex, LN₂</td>
<td>Vortex, LN₂</td>
</tr>
<tr>
<td>Electrical temperature control of the measuring cell</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
</tr>
<tr>
<td>Force change (ramp, step at each new segment)</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
</tr>
<tr>
<td>Force modulation</td>
<td>✓</td>
<td>□</td>
<td>✓</td>
</tr>
<tr>
<td>Single/double DIL</td>
<td>✓ / □</td>
<td>✓ / □</td>
<td>✓ / □</td>
</tr>
<tr>
<td>Automatic sample length detection</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
</tr>
<tr>
<td>Softening Point detection</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
</tr>
<tr>
<td>Density Determination</td>
<td>✓</td>
<td>□</td>
<td>✓</td>
</tr>
<tr>
<td>c-DTA*</td>
<td>✓</td>
<td>□</td>
<td>**</td>
</tr>
<tr>
<td>RCS (Rate-Controlled Sintering)</td>
<td>✓</td>
<td>□</td>
<td>✓</td>
</tr>
<tr>
<td>Identify</td>
<td>✓</td>
<td>□</td>
<td>✓</td>
</tr>
<tr>
<td>Evolved gas analysis (coupling with GC-MS/QMS and/or FT-IR) – for SiC furnace</td>
<td>□</td>
<td>□</td>
<td>□</td>
</tr>
</tbody>
</table>

* DIL 402 Expedis® Supreme HT with adapter for standard furnaces
** Not above 2000°C, only by thermocouple operation

Both instrument models work on the basis of DIN 51045, ASTM E228, ASTM D696 or DIN EN 821.

- Included in standard configuration
- Optional
- N/A Not applicable
## Technical Specifications

### DIL 402 Expedis® Supreme, Supreme HT and Select

<table>
<thead>
<tr>
<th>Design</th>
<th>Pushrod dilatometer, single or dual system</th>
</tr>
</thead>
<tbody>
<tr>
<td>Furnaces</td>
<td>Different types, interchangeable (for Supreme HT partially with adapter): steel, copper, SiO₂, SiC (optional furnace for fast cooling available), Rh; graphite (only for Supreme version)</td>
</tr>
</tbody>
</table>
| Heating rates     | Depending on furnace type:  
- Steel, copper, fused silica, silicon carbide: 0.001 ... 50 K/min  
- Graphite: 0.001 ... 100 K/min |
| Cooling systems   | Depending on furnace: Vortex, LN₂-device, air compressor |
| Sample holder systems | SiO₂, Al₂O₃, graphite (Supreme version), user interchangeable  
All sample holders are available as  
- Single system (one pushrod)  
- System with two pushrods usable in dual or differential mode  
- Al₂O₃ tension sample holder*  
SiO₂ and Al₂O₃ sample holders can be purchased as tube or rod design |
| Sample dimensions | Max. length: 52 mm (graphite furnace: 25 mm)  
Diameter (single): standard 12 mm, optional 19 mm  
Diameter (dual): 8 mm |
| Automatic sample length determination | Yes, in expansion mode |
| Displacement system | NanoEye |
| Temperature accuracy / precision / resolution | 1 K / 0.1 K / 0.001 K |
| Thermal stability (isothermal) | ± 0.02 K |
| Temperature calibration | Displacement method (by using metal references and protective disks)  
or via c-DTA® (optional for Select version; incl. endo/exothermal effects) |
| Measuring range | ± 25000 µm (Supreme version)  
± 10000 µm (Select version) |
| ΔL Resolution | 0.1 nm (Supreme version)  
1 nm (Select version) |
| ΔL/L₀ Repeatability | 0.001 %, absolute value |
| ΔL/L₀ Accuracy | 0.002 %, absolute value |
| Force range (load at the sample) | 10 mN ... 3 N |
| Change of force | Supreme version: various options, incl. modulated forces  
Select version: changeable per segment (constant & ramp) |
| Force resolution | 0.001 mN |
| Gas atmosphere | Inert, oxidizing, reducing, vacuum |
| Gas control | MFC  
- Standard: 1 x protective gas  
- Optional: 1 x protective gas, 2 x purge gas |
| Oxygen Trap System (OTS*) | Optional, for single and for dual sample holder systems |
| Software | Windows 7 32/64 bit Professional®, Enterprise® and 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 as well as the temperature range when using the copper furnace.
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.
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.

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

NETZSCH®
www.netzsch.com