LINSEIS

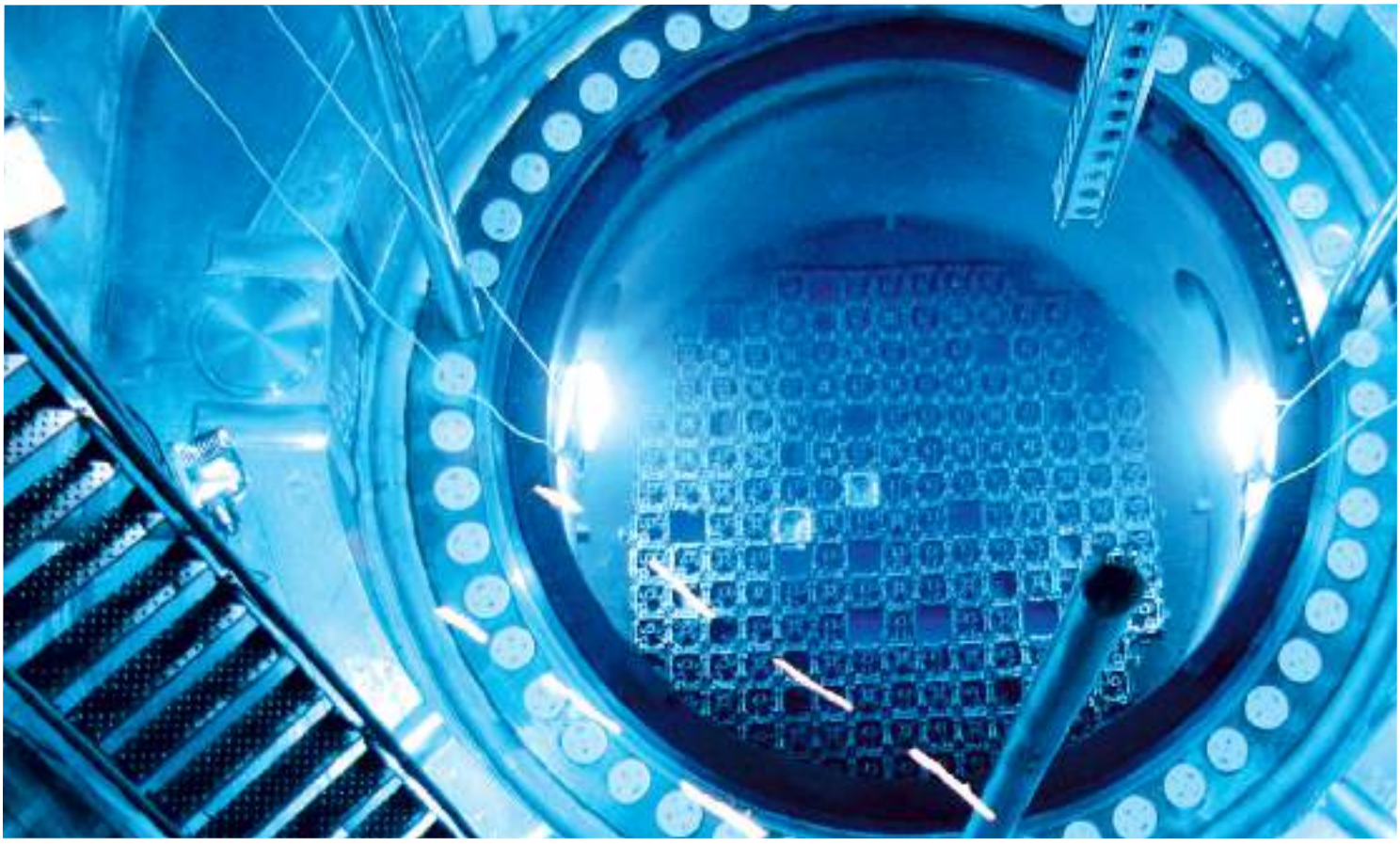
THERMAL ANALYSIS

# NUCLEAR

DIL

STA

LFA



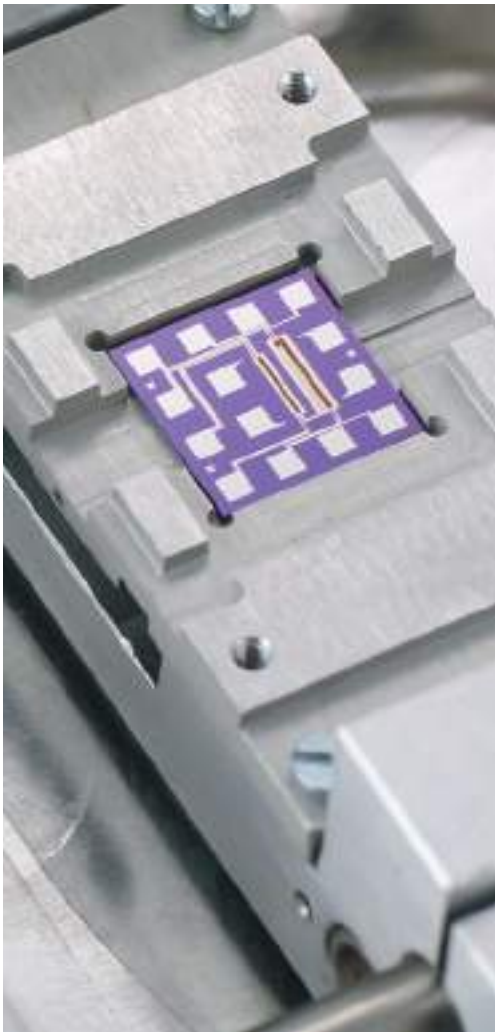
Since 1957 LINSEIS Corporation has been delivering outstanding service, know how and leading innovative products in the field of thermal analysis and thermo physical properties.

We are driven by innovation and customer satisfaction.

Customer satisfaction, innovation, flexibility and high quality are what LINSEIS represents. Thanks to these fundamentals our company enjoys an exceptional reputation among the leading scientific and industrial organizations. LINSEIS has been offering highly innovative benchmark products for many years.

The LINSEIS business unit of thermal analysis is involved in the complete range of thermo analytical equipment for R&D as well as quality control. We support applications in sectors such as polymers, chemical industry, inorganic building materials and environmental analytics. In addition, thermo physical properties of solids, liquids and melts can be analyzed.
LINSEIS provides technological leadership. We develop and manufacture thermo analytic and thermo physical testing equipment to the highest standards and precision. Due to our innovative drive and precision, we are a leading manufacturer of thermal Analysis equipment.
The development of thermo analytical testing machines requires significant research and a high degree of precision. LINSEIS Corp. invests in this research to the benefit of our customers.

**Claus Linseis**
Managing Director



## German engineering

The strive for the best due diligence and accountability is part of our DNA. Our history is affected by German engineering and strict quality control.

## Innovation

We want to deliver the latest and best technology for our customers. LINSEIS continues to innovate and enhance our existing thermal analyzers. Our goal is constantly develop new technologies to enable continued discovery in Science.

# THERMAL ANALYSIS OF NUCLEAR MATERIALS

## Special field of application: Nuclear materials

Since the 1950s nuclear energy is the worldwide most important energy source around the world. With their advantage of clean and cheap power supply, core reactors were undergoing a continuous global improvement during the last 50 years. Meanwhile the reactors of the 4th generation such as very high temperature reactors (VHTR) or sodium cooled fast reactors (SFR) as well as the unique molten salt reactor (MSR) are currently under development and will be the future for nuclear energy.

Due to the research that is done in that field, there is a need of analytical equipment and especially instruments for thermal analysis. Of course these special applications and safety requirements need a lot of modifications of the standard devices, that makes Linseis become the worldwide leader in thermal analysis of nuclear materials as we are the most flexible and most experienced player on that market.

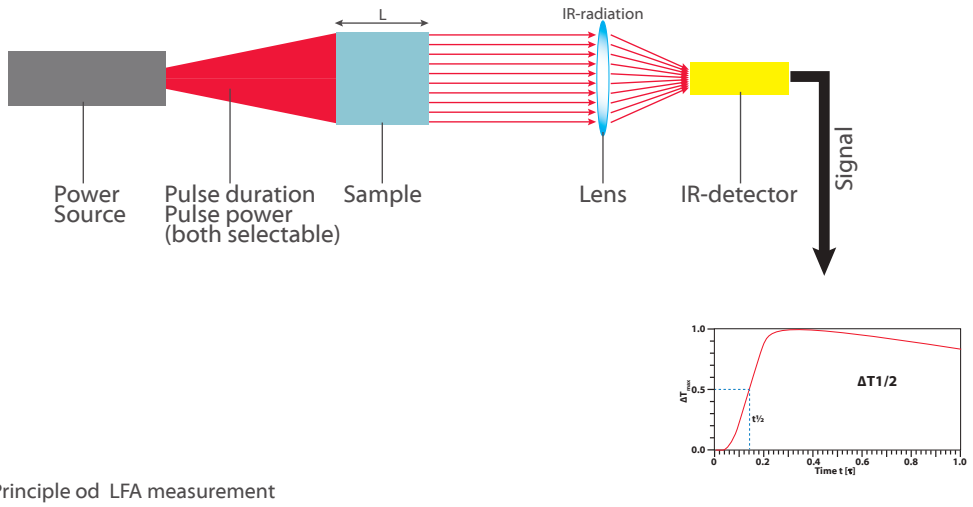
# LFA

## Standard Test Method for Thermal Diffusivity by the Flash Method

A small, thin disc specimen is subjected to a high intensity short duration radiant energy pulse. The energy of the pulse is absorbed on the front surface of the specimen and the resulting rear face temperature rise is recorded. The thermal diffusivity value is calculated from the specimen thickness and the time required for the rear face temperature rise to reach certain percentages of its maximum value.

Out of density and Cp the thermal conductivity can be calculated using the following formula:

$$\lambda(T) = a(T) \cdot c_p(T) \cdot \rho(T)$$



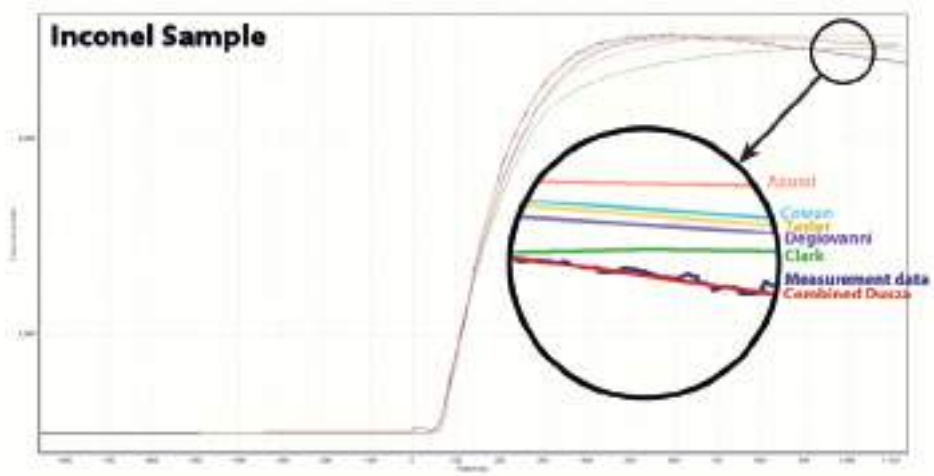

Principle od LFA measurement

## Calculation of thermal diffusivity

- Determine the baseline and maximum rise to give the temperature difference, $\Delta T_{max}$
- Determine the time required from the initiation of the pulse for the rear face temperature to reach $\Delta T_{½}$ . This is the half time, $t_{½}$.
- Calculate the thermal diffusivity, a, from the specimen thickness, L squared and the half time $t_{½}$, as follows:

$$\alpha = 0.13879\ L^2/t_{½}$$

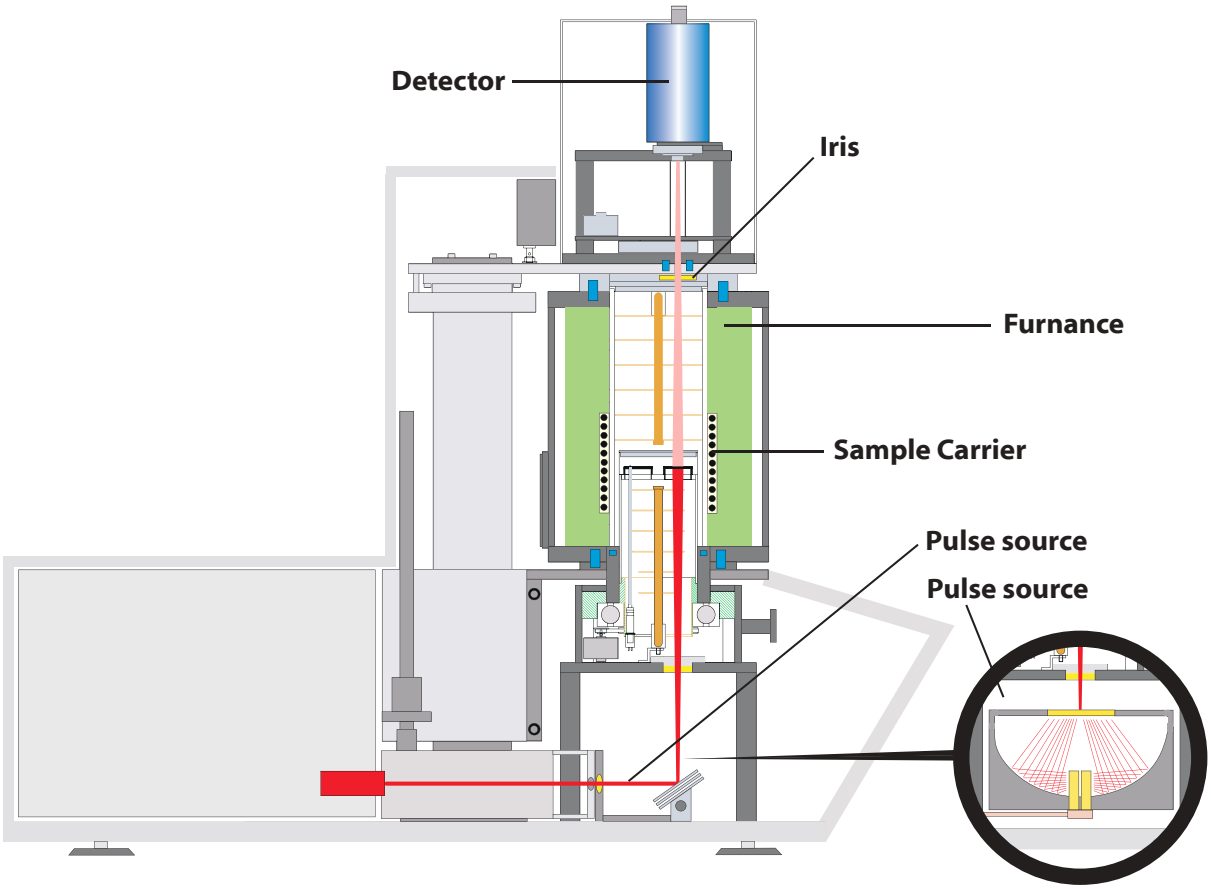
## Combined solution of the simultaneous heat loss and finite pulse corrections with the laser flash method




Inconel Sample
Model Comparison-Demonstrating superior performance of the combined model versus classic models of: Azumi, Clark, Cowan, Degiovanni and Tayler

## Conclusion

The combined model method with nonlinear parameter estimation has been proven for more than 100 samples. In all cases it worked reliably and its results gave the correct adiabatic, finite pulse, and/or heat loss corrected values. The two main advantages of the method are that no operator choice between the different models and correction is necessary, and the fit can be checked by plotting the model curve.


Detector
Iris
Furnance
Sample Carrier
Pulse source
Pulse source

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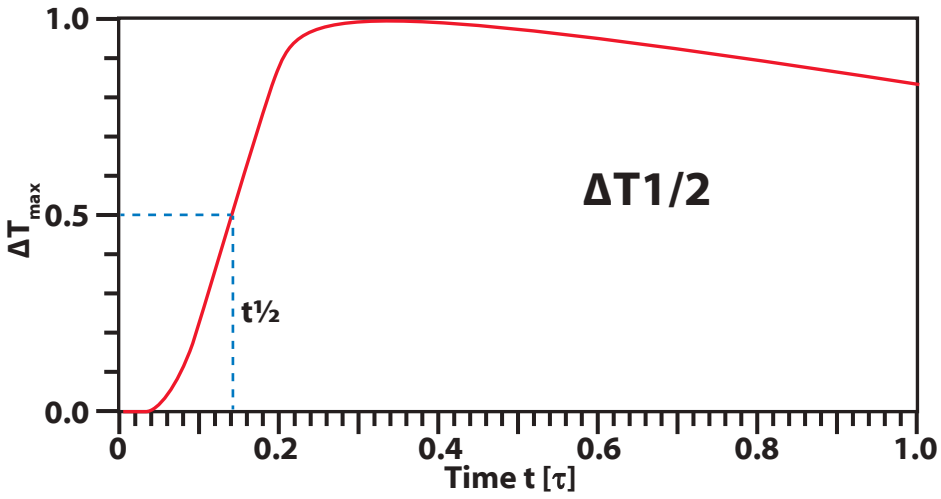



## For Special setup for radioactive or toxic samples

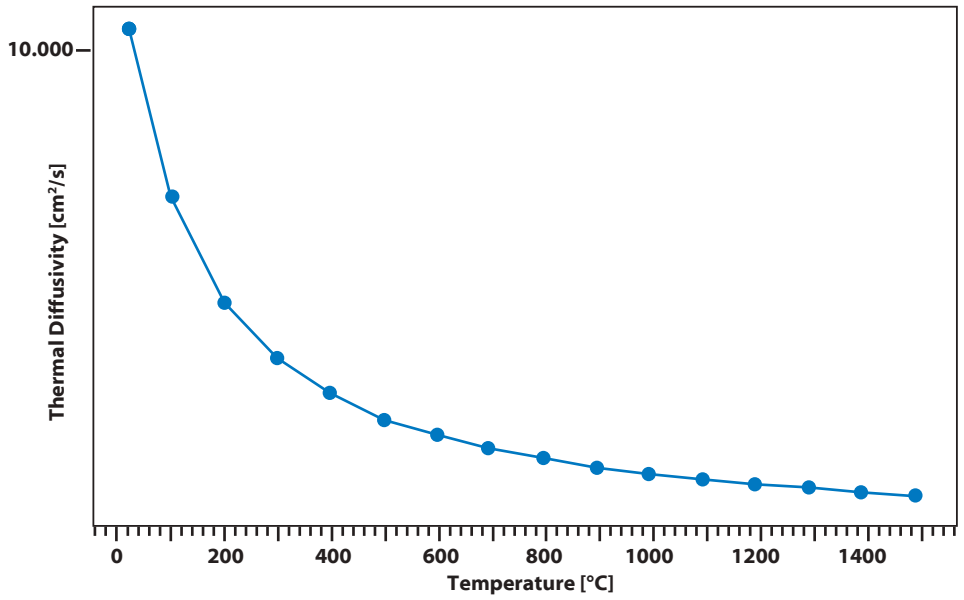
- Measurement unit separated from control electronics and laser
- Laser connected by fibre glass optical cable
- Measurement unit can be placed in hood or glovebox
- Maintenance, operation and setup possible in glovebox

# TECHNICAL SPECIFICATIONS

| | LFA 500 IR | LFA 1000 | LFA 1000 HT |
|---|---|---|---|
| **Sample dimesnions** | ø 10 / 12.7 / 25.4 mm, 0.1 to 6 mm thick<br><br>10 / 20 mm; 0.1 up to 6mm thick | | |
| **Samples** | solids, liquids, powders. pastes | solids, liquids, powders. pastes | solids, liquids, powders. pastes |
| **Sample robot** | up to 6 samples | up to 6 samples | up to 3 samples |
| **Vacuum** | depends on model up to $10^{-4}$mbar | | |
| **Atmosphere** | inert, oxidizing or reducing | | |
| **Measuring range Thermal Diffusivity** | 0.01 up to 1000 $mm^2/s$ | 0.01 up to 1000 $mm^2/s$ | 0.01 up to 1000 $mm^2/s$ |
| **Measuring range Thermal Conductivity** | 0.1 to 2000 W/(m·K) | 0.1 to 2000 W/(m·K) | 0.1 to 2000 W/(m·K) |
| **Pulse source** | Flash lamp | Flash lamp | Flash lamp |
| **Pulse energy** | 10 J/pulse | 25 J/pulse | 25 J/pulse |
| **Pulse energy & pulse duration adjustment** | yes | yes | yes |
| **Pulse length adjustment** | software adjustable | software adjustable | software adjustable |
| **Sensor type** | InSb / MCT | InSb / MCT | InSb / MCT |
| **Furnace model** | **IR-furnace**<br>RT up to 500°C<br>RT up to 1000°C<br><br>**Cryo-Furnace**<br>-125 up to 600°C | **Resistance heater**<br>RT up to 1250°C<br>RT up to 1600°C | **Graphite**<br>RT up to 2000°C<br>RT up to 2800°C |

# APPLICATIONS

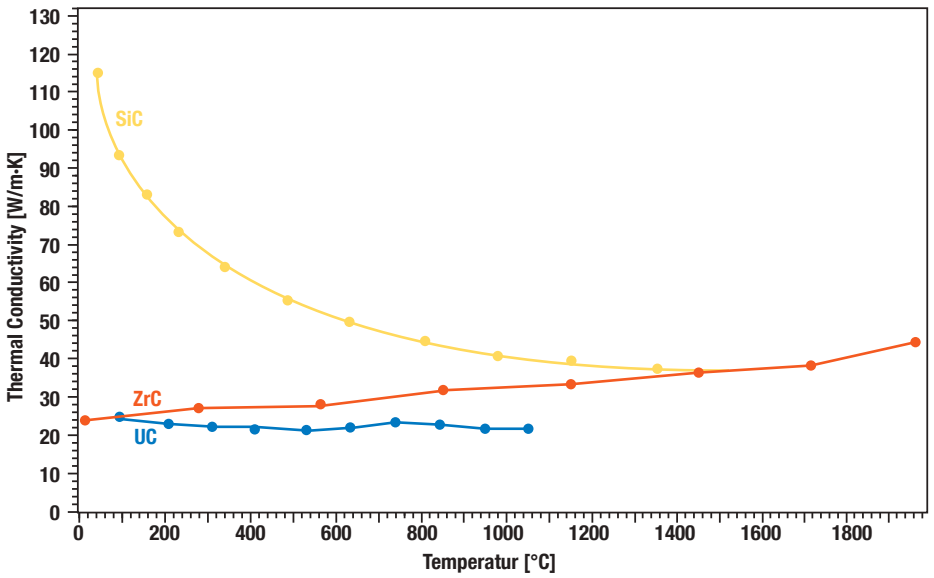
## Graphite standard




The curve shows the thermal diffusivity data of a graphite standard from NIST that was measured with a special LFA with fibre-optical connected laser and separated electronics. The results are matching with the literature values and accuracy and power of the laser are completely identical with the standard instrument.

Graphite is one of the most important materials in reactor construction and due to its high thermal conductivity and temperature stability it offers various purposes it can be used for.

## Thermal Conductivity of three carbide ceramics




Three different types of carbides were measured by LFA to determine the thermal conductivity. One of the samples has been the uranium derivative that showed a considerable low value of around 25 W/mK. ZrC shows a increasing trend that has a electronic dominant character, whereas the SiC shows a more or less common decrease shape.

# DILATOMETRY

## Technical Expansion Coefficient

The technical expansion coefficient can be calculated as follows:

$$\alpha_{tech}(k) = \frac{1}{L_0} \quad \frac{\Delta L_k - \Delta L_0}{T_k - T_0} \qquad (k=1....n)$$

for every measured point k.

$L_0$ is the sample length at 20°C, $\Delta L_0$ the change in length at 20°C (linear extrapolated out of the first data points), $\Delta L_k$ the according length change at the temperature Tk.

## Measuring thermal expansion of radioactive samples

The standard instrument for measuring the thermal expansion coefficient (CTE), the Dilatometer, detects the change in length of any sample by LVDT detector systems. For toxic and radioactive samples, it was necessary to remove all electronics and build a special furnace that is separated from the detector. Beside this, the design was also changed to a more accessible setup for maintenance and mechanical changes under glovebox or fume hood.

Another method for CTE determination is the contact free optical dilatometer. It can be used with any atmosphere and the sample does not see any force. For less x-ray emitting samples it can be a very useful instrument, however it can also be placed within a glovebox or fume hood.

## Measuring systems

The LINSEIS L75 Dilatometer can be used with different measurement systems for various applications, even for measuring powders and pastes, offering a broad range in temperature and field of application.

**Quartz measuring system ø 7/12 mm**

**$Al_2O_3$ measuring system contact free**

**$Al_2O_3$ measuring system standard**

**Adapter for powders and pastes**

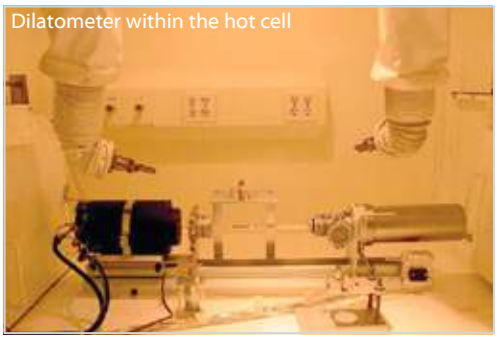
**Quartz measuring systemfor large samples ø 20mm**

# TECHNICAL SPECIFICATIONS

| | L 75 Horizontal | L75 Vertical | L75 Laser | L74 Optical |
|---|---|---|---|---|
| **Temperature range** | -180 up to 2000°C | -180 up to 2800°C | -180 up to 1000°C | -100 up to 2000°C |
| **Atmospheres** | Inert, oxidizing, reducing | Inert, oxidizing, reducing, vacuum | Inert, oxidizing, reducing, vacuum | Inert, oxidizing, reducing, vacuum |
| **Resolution** | 1nm | 1nm | 0.3nm | 1µm |

## Thermal expansion Measurements

We find that the Coefficient of Thermal Expansion (CTE) of super-invar is sensitive to the level of irradiation exposure.


Dilatometer within the hot cell

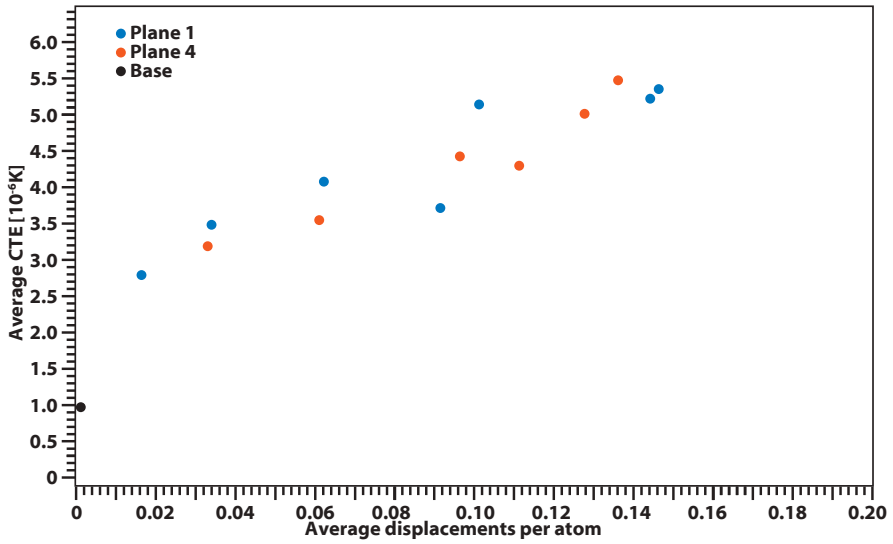
## Customer Application from Brookhaven:

The CTE of invar is dependent on the level of irridiation exposure.

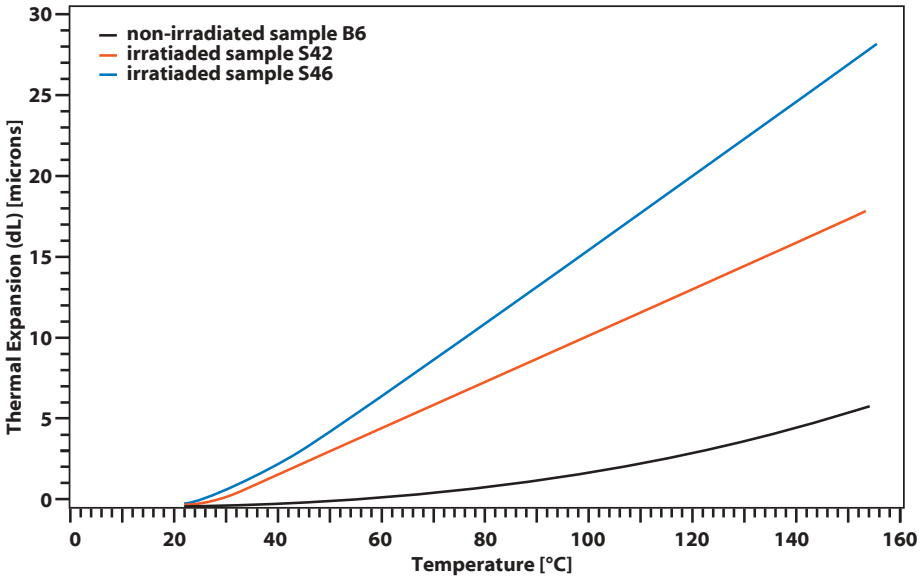
The experiments and studies were performed using our L75 horizontal as a special version in a glovebox.

# APPLICATIONS

## Super-invar



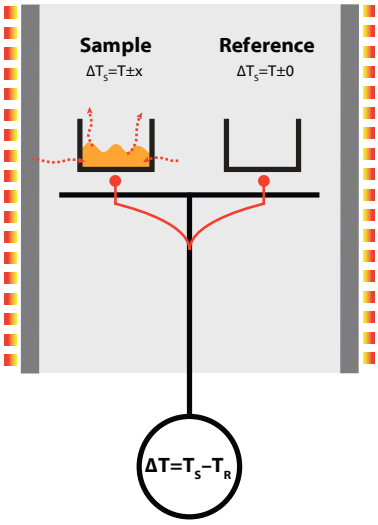

Harold G. Kirk

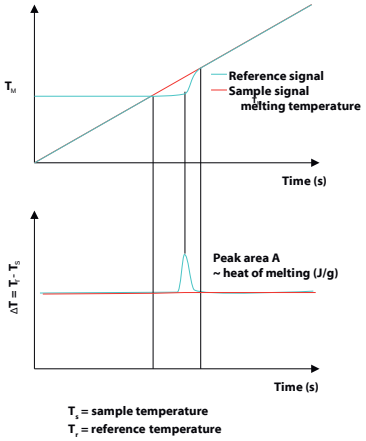

non-irradiated sample B6
irratiaded sample S42
irratiaded sample S46
Thermal Expansion (dL) [microns]
30
25
20
15
10
5
0
0
20
40
60
80
100
120
140
160
Temperature [°C]

# STA

# DSC- True Heat Flow measurement

## Quantitative DSC-signal




### Differential Scanning Calorimetry (DSC)

"A technique in which the difference in energy input into a substance and a reference material is measured as a function of temperature, while the substance and reference material are subjected to a controlled temperature program."

### Differential Signal

The differential signal is displayed as a baseline. Effects, for example the melting of a metal, can be observed as a peak. The area of the peak gives the amount of enthalpy and the direction of the peak indicates the way of heat flux – endothermic (down) or exothermic (up).

### Temperature vs. Time

During an effect like a reaction, decomposition or phase transition, a temperature difference (heat flux difference) between the sample and the reference crucible can be measured by means of a thermocouple.

## MEASURABLE PROPERTIES

- Mass change as % and mg
- Rate controlled mass loss
- Evaluation of mass loss
- Residue mass evaluation
- Compositional analysis

- Enthalpy
- Endo- / Exo- thermic
- Phase transformation
- Melting point
- Glass point
- Crystallinity

- Thermal stability
- Oxidation stability
- Purity
- Solidus / Liquidus relationship
- Product identification

# SENSORS

Our STA can be equipped with an unmatched amount of different user exchangeable TG-DSC, TG-DTA or TG sensors.

Each sensor is available with different thermo-couples to provide the highest sensitivty for your desired temperature range.

## TG-DSC

DSC Heat flux

3D-Calvet-DSC sensor

0.12ml

0.12ml

custom volume

Different crucibles available / broad variety

- $Al_2O_3$
- Platinum
- Aluminium
- others

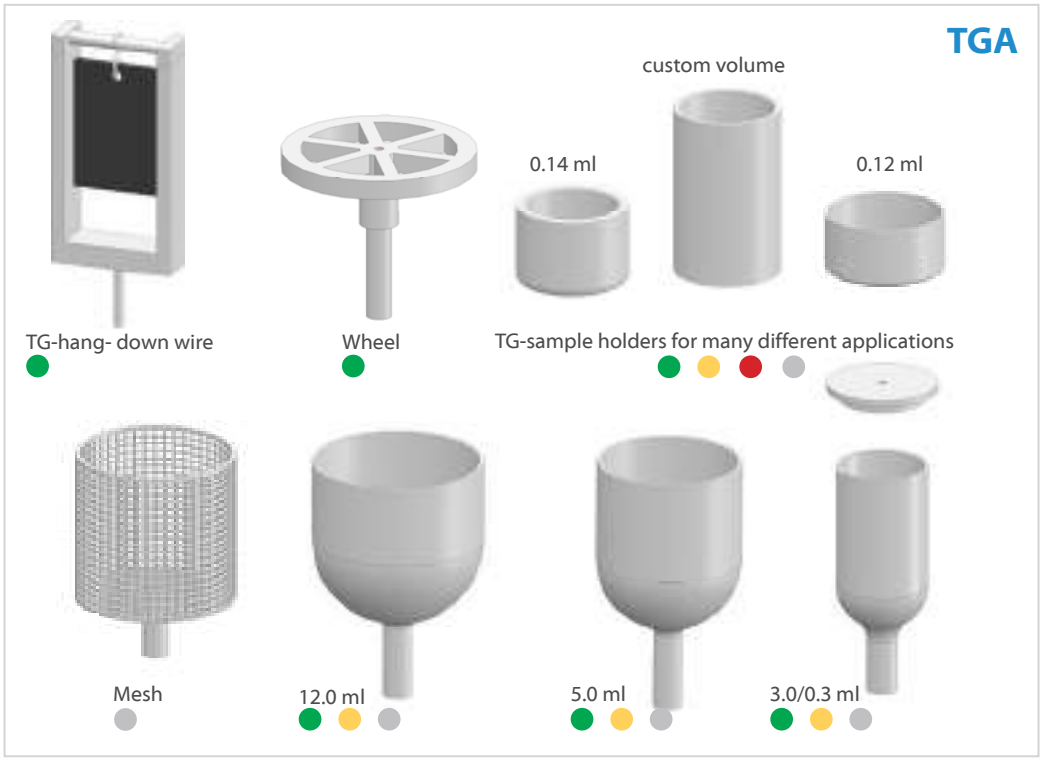
## TG-DTA

DTA-sensor

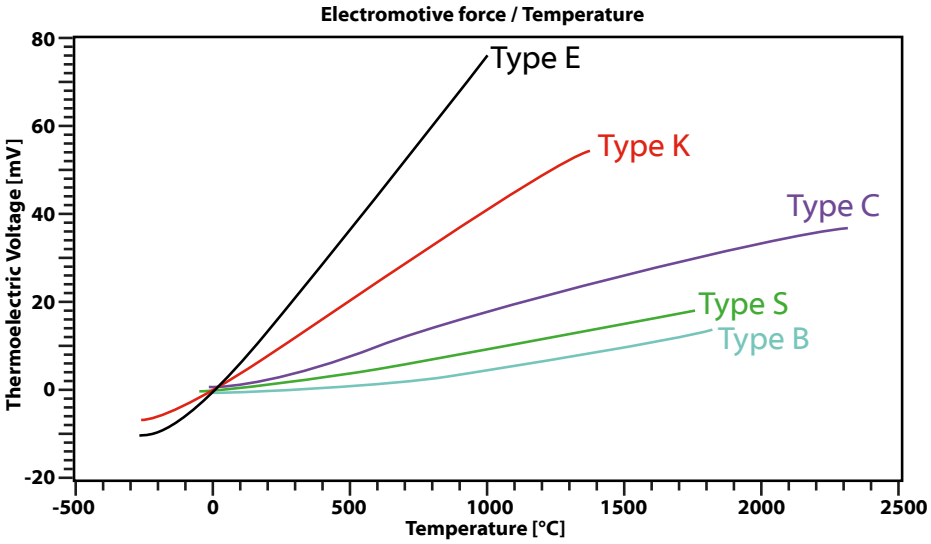
Cap

0.3ml or
custom volume

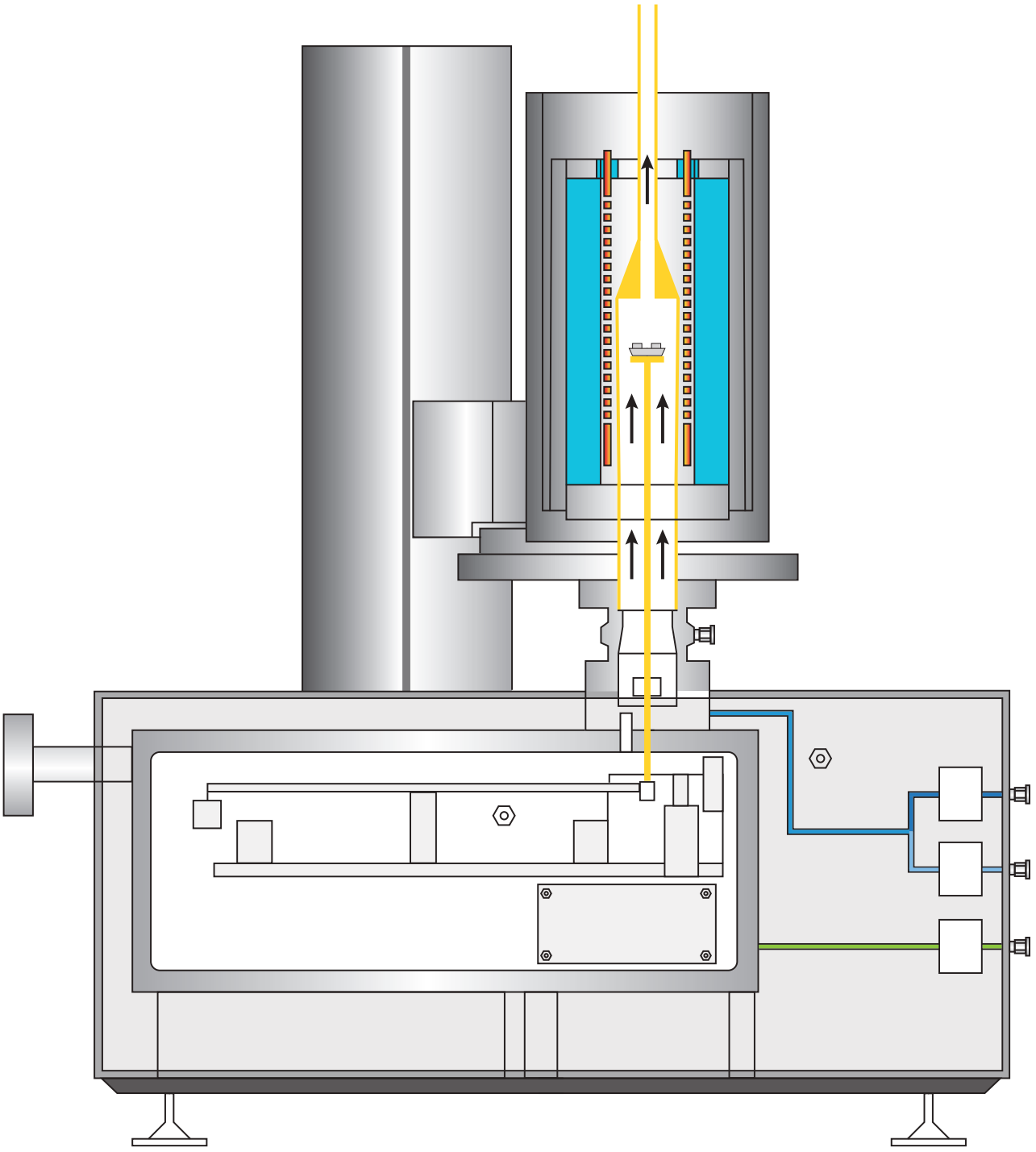
Crucible




# Best possible sensitivty for your application



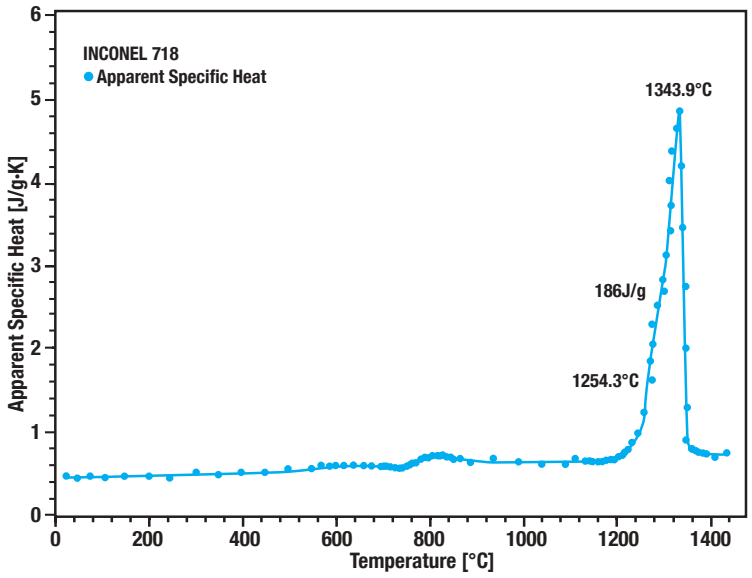

All sensors available with the thermocouples illustrated. LINSEIS Senosor combinations cover the broadest temeprature range in the market (-180 up to 2400°C).



# TECHNICAL SPECIFICATIONS

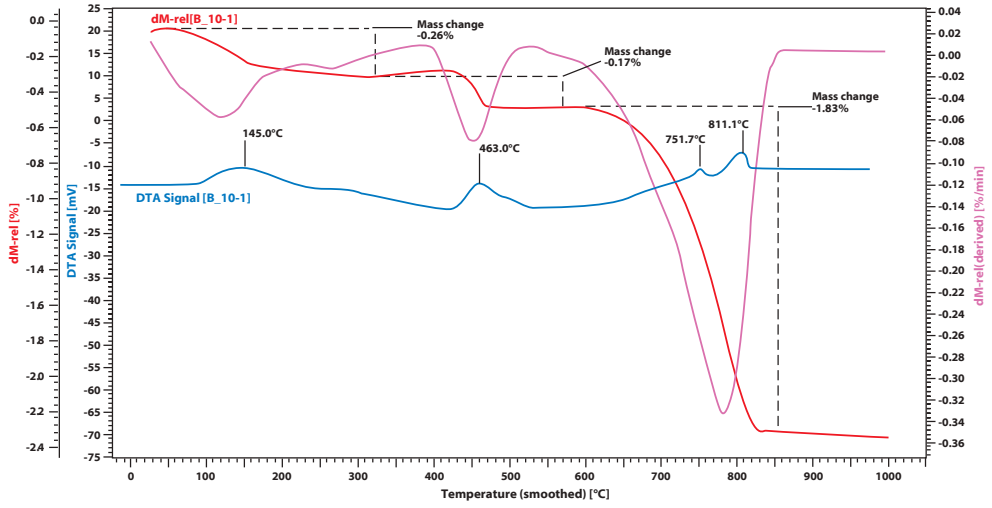
| | STA PT 1000 | STA PT 1600 | | | STA HP/2 | |
|---|---|---|---|---|---|---|
| | | | | | | |
| **Temperature range** | RT up to 1000°C | -150 up to 2400°C | | | RT up to 1100/ 1400°C/1600°C/1800°C | |
| **Vacuum** | optional $10^{-2}$ mbar | $10^{-5}$ mbar (depends on vacuum pump) | | | up to $10^{-4}$mbar | |
| **Pressure** | | up to 5 bar (optional) | | | up to 150 bar custom solution on request | |
| **Heating rate** | 0.01 up to 100°C/min | 0.01 up to 100°C/min (depends on furnace) | | | 0.01 up to 100°C/min (depends on furnace) | |
| **Temperature precision** | 0.01°C | 0.01°C | | | 0.05°C | |
| **Sample robot** | optional 42 / 84 | optional 42 | | | - | |
| **TG** | | | | | | |
| **Resolution** | 0.1 µg | 0.025 µg | 0.1 µg | 0.1 µg | 0.1 µg | 0.1 µg |
| **Sample weight** | 5 g | 5 g | 25 g | 35 / 50 g | 15 / 25 g | 35 / 50 g |
| **Measuring range** | 25 / 2500 mg | 25 / 2500 mg | 25 / 2500 mg | 35000 mg | 25 / 2500 mg | 35000 mg |
| **DSC** | | | | | | |
| **DSC-sensors** | E / K / S | E / K / S / B / C | | | E / K / S / B / C | |
| **DSC resolution** | 0.3 / 0.4 / 1µW | 0.3 / 0.4 / 1 / 1.2 µW | | | 0.3 / 0.4 / 1 / 1.2 µW | |
| **Calorimetric sensitivity** | approx. 4 / 6 / 17.6 µW | approx. 4 / 6 / 17.6 / 22.5 µW | | | approx. 4 / 6 / 17.6 / 22.5 µW | |
| **DTA** | | | | | | |
| **DTA-resolution** | 0.05 µV | 0.05 µV | | | 0.05 µV | |
| **Sensitivity** | 1.5 µV/mW | 1.5 µV/mW | | | 1.5 µV/mW | |
| **DTA-measuring ranges** | 250 / 2500 µV | 250 / 2500 µV | | | 250 / 2500 µV | |

## INCONEL 718



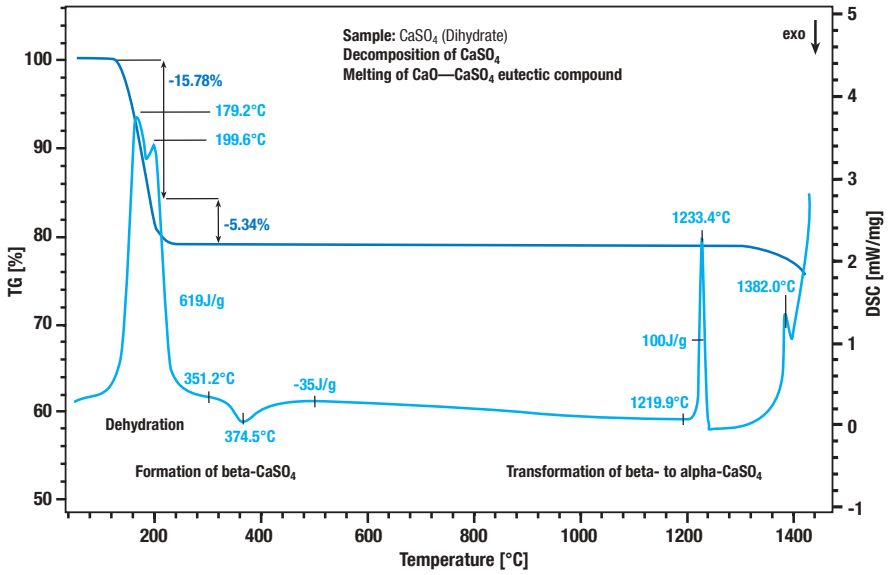

The curve shows the specific heat of Inconel 718 during heating. The steel can be used to produce super invar steal that can be hardened by irradiation. Out of the specific heat curve, the solidus and liquidus temperature at 1254°C and 1343°C as well as the melting enthalpy of 186J/g can be determined.

## Cement




The main parts of cement are tri calcium silicate, di calcium silicate and tri calcium aluminates. After putting on the cement with water different hydrates slowly form. The absorbed water evaporates first, then hydrates of the calcium silicate decompose and at 570°C the hydroxides of calcium, magnesium and aluminum follow. Subsequently, calcium carbonate $CO_2$ splits off.

## Decomposition of gypsum and quartz sand




Typical Applications for Gypsum and quartz sand are plaster and mortar. The content of gypsum in the sample illustrates a two-step release of $H_2O$ from $CaSO_4 \cdot 2H_2O$ (dihydrate) into $CaSO_4 \cdot ½H_2O$ (halfhydrate) and finally into $CaSO_4$ (anhydrite). This requires an entire energy of 121.6 J/g. The Quantitative analysis (TG-DSC Sensor) reveals that the sample contained 23.4% of pure dihydrate. Between approx. 300°C and 450°C, the exothermic formation of β-CaSO4 with a released energy of 18.32 J/g occurred. The endothermic effect at a temperature of 574.9°C is due to the structural $\alpha \rightarrow \beta$ transition of quartz (crystalline $SiO_2$).

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**Services:** Service Lab, Calibration Service

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