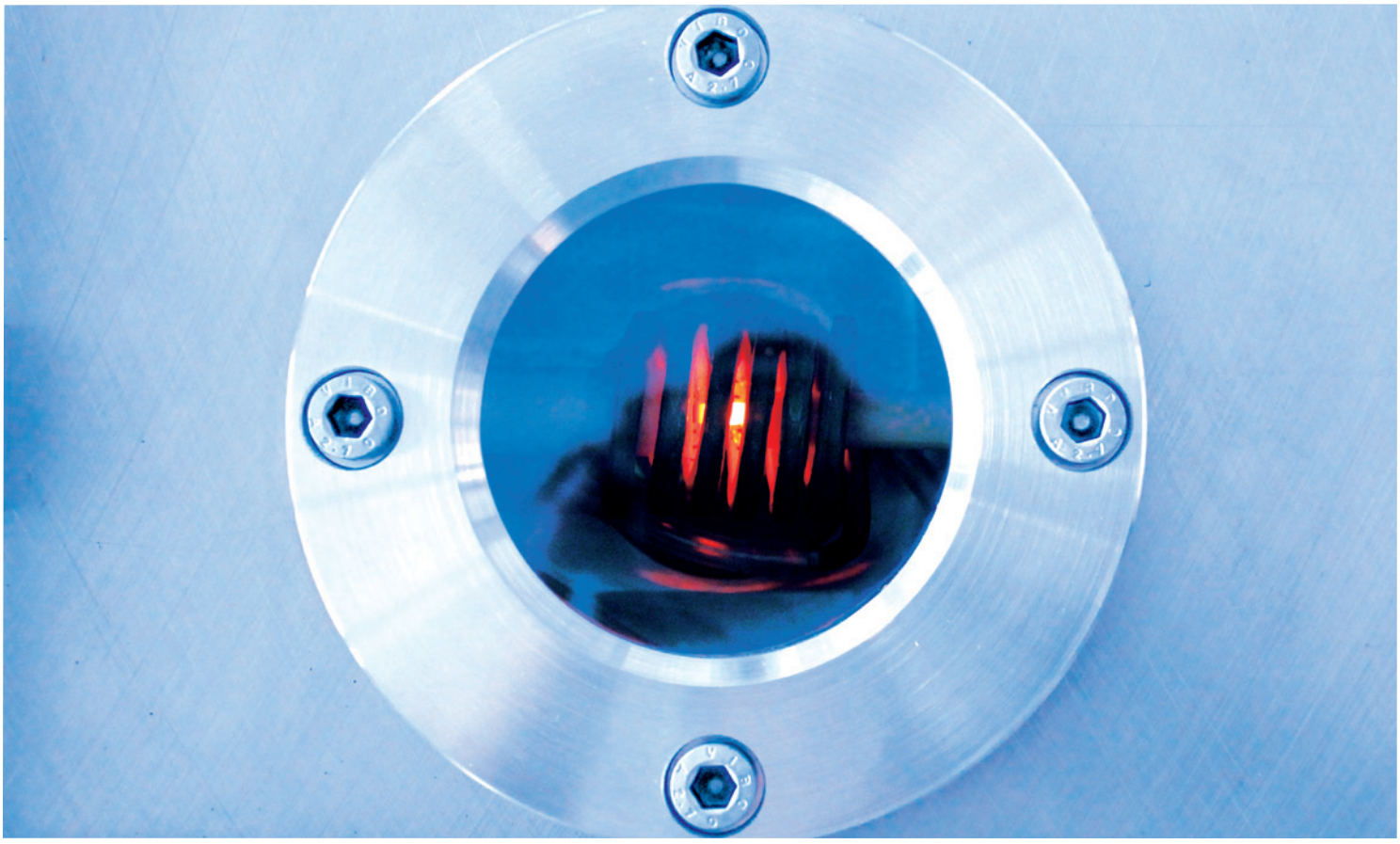
LINSEIS

THERMAL ANALYSIS

Quenching & Deformation Dilatometer

DIL L78 Q/D/T

DIL L78 Q



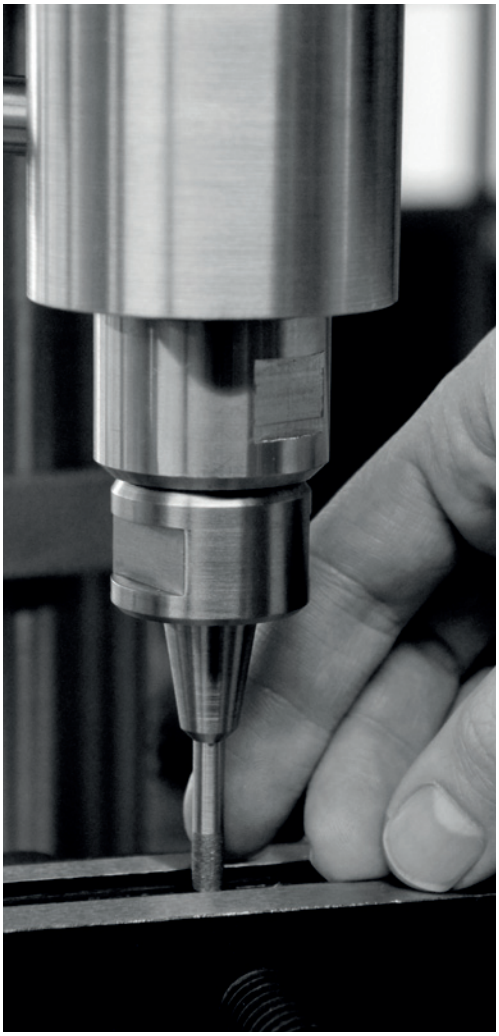
Since 1957 LINSEIS Corporation has been delivering outstanding service, know how and leading innovative products in the field of thermal analysis and thermo physical properties.

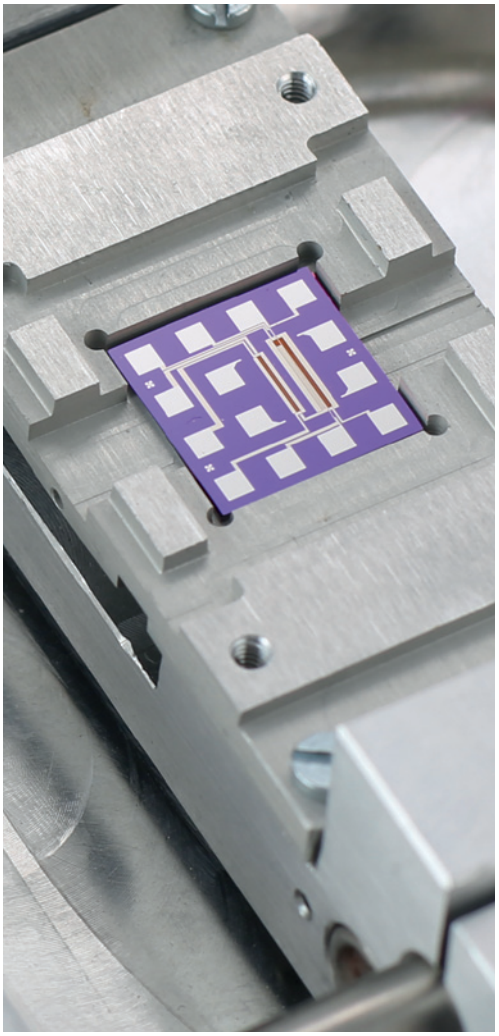
We are driven by innovation and customer satisfaction.

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.

QUENCHING & DEFORMATION DILATOMETER

up to 4500 K/s

LINSEIS L78/Q/D/T

- The Quenching and Deformation Dilatometer L78 RITA is especially suitable for the determination of deformation paramenter and of TTT, CHT, CCT and DCCT diagrams.
- The special induction furnace allows heating and cooling at controlled speeds up to 4000 K/s.
- Different cooling gases can be used, e.g. argon, helium and nitrogen. The system complies with ASTM A1033.
- All critical parameters such as heat up and cool down speed, gas control and safety features are software controlled.
- The professional software LINSEIS TA-WIN operates exclusively under the Microsoft© operation system. All routine (creation of CHT/CCT/DCCT/TTT diagrams) and demanding applications are solved by the unique Software package that comes with the instrument.
- Export functions in ASCII-format as well as graphic output are available.

QUENCHING DILATOMETER

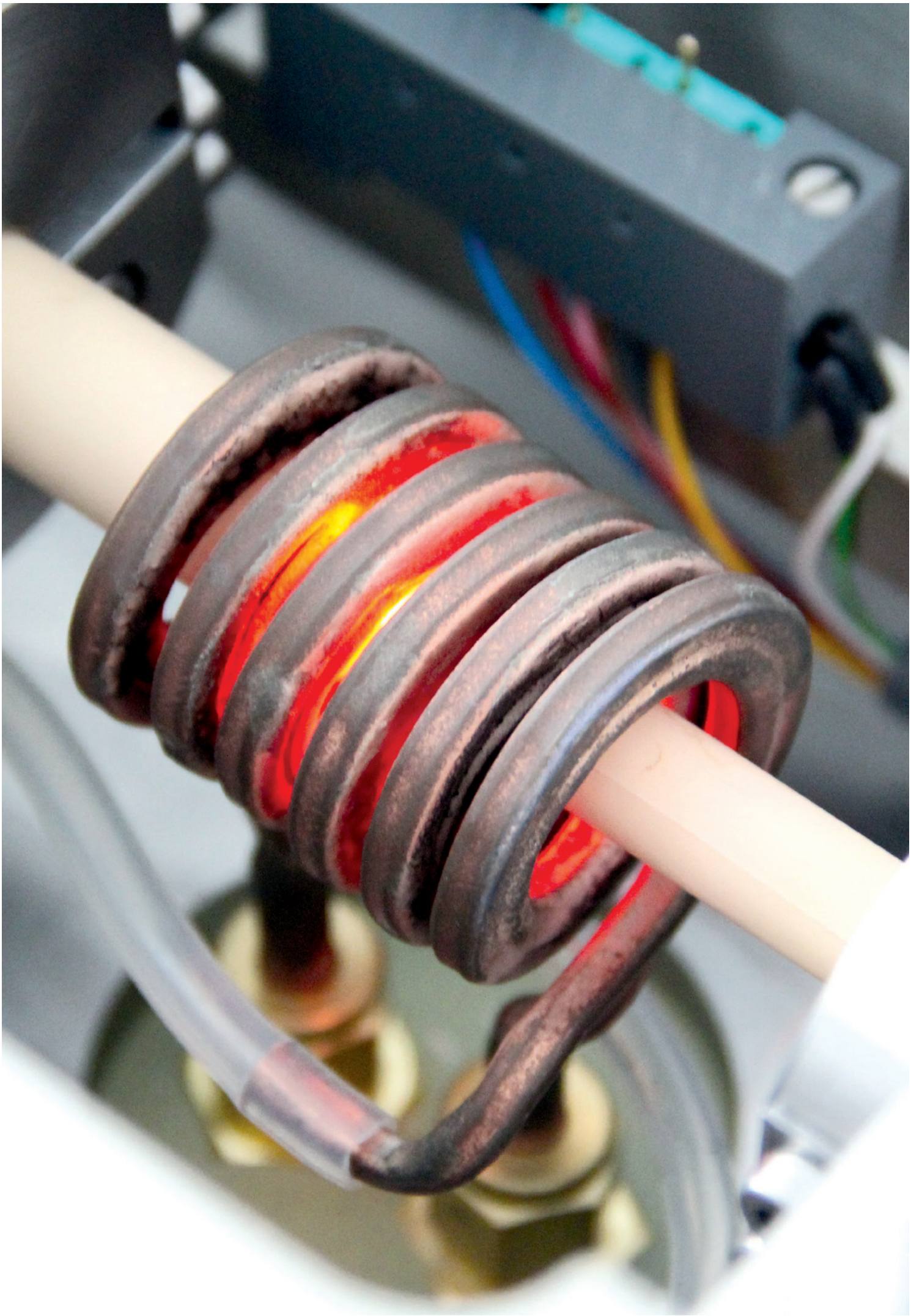
LINSEIS L78/Q

- The used linerar actuator machanical system makes it possible to achieve deformation rates from 0.01 up to 100 mm/s in single or multiple hits.

System configurations

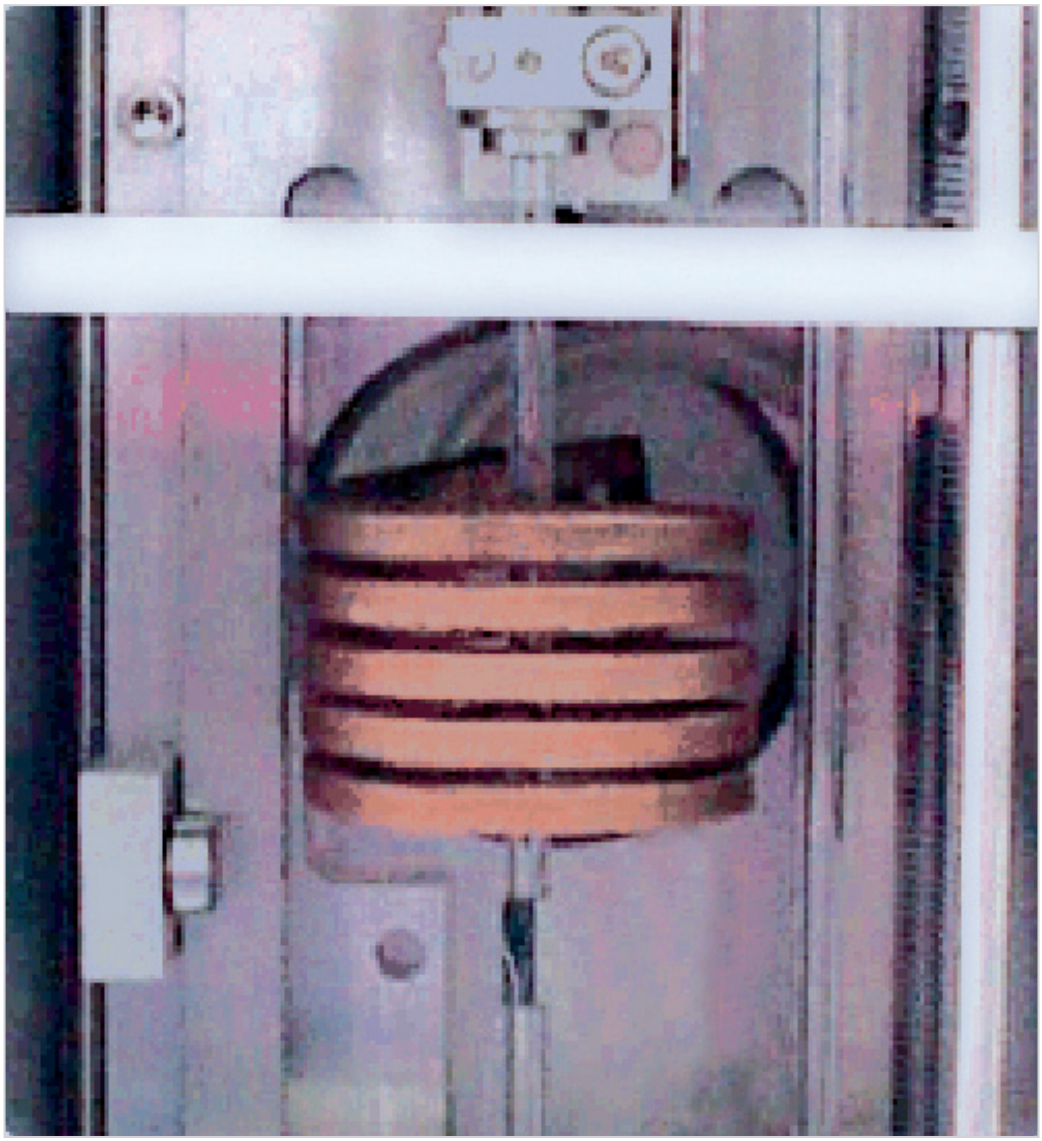
L78 Q/D/T Dilatometer Modules

- CTE option (dilatation)
- Cryogenic option
  (temperature range: –150°C)
- DSC option (up to 1100°C / 1450°C on request)
- ODS optical displacement sensor
- Deformation mode
- Tension mode



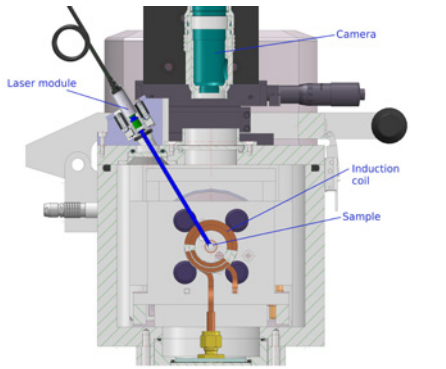
ADVANTAGES

- The instrument can perform measurements under vacuum, inert, oxidized, reduced atmospheres from 150 (low temperature option) up to 1000°C and room temperature up to 1600°C in one run.
- The unique heating and cooling arrangement enable very fast controlled heat up and cool down speeds of up to 4500 / 3000K/s.
- With the optional susceptor non metallic samples can me analyzed.
- This special Quenching dilatometer is especially designed for the determination of continuous cooling / heat up CHT, CCT and isotherm TTT- diagrams.



DETERMINATION OF CONVERSION TEMPERATURES OF STEEL SAMPLES WITH THE HELP OF LASER SPECKLES

Measuring the temperature dependent change in length can be used to determine the temperature at which a structural transformation takes place in steel samples. With the laser speckles method, the sample surface is illuminated a laser and observed from above with a camera. At the same time, the sample is heated with the help of an induction coil. To filter the heat radiation and the light that emitted from glowing sample, a blue laser is used and appropriate filters are attached in front of the camera.



Schematic of the setup

Measurement

During the measurement, the sample is heated inductively and at the same time the speckle patterns generated by the laser are observed with the camera. Depending on the speed of the measurement, up to 50 frames per second can be recorded.

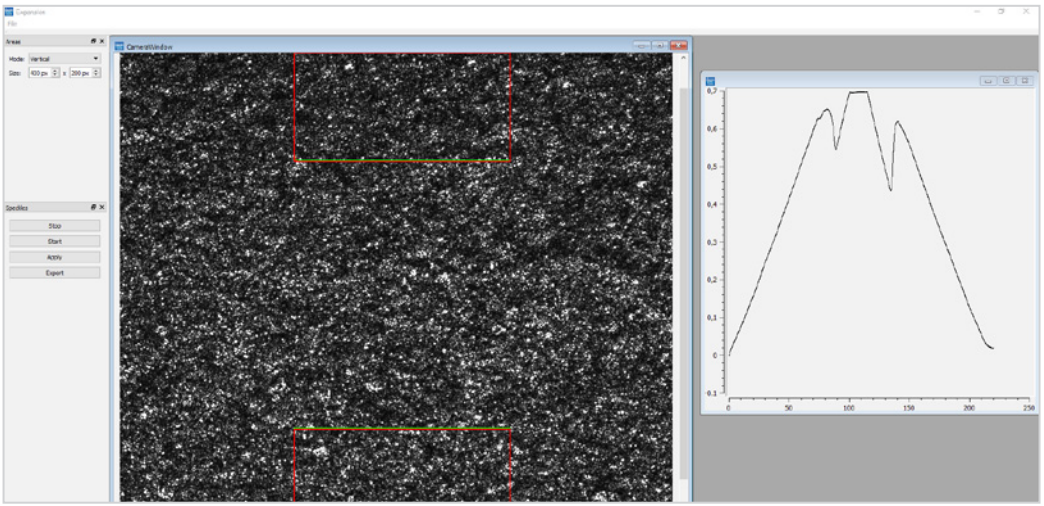
Evaluation

The camera generates sequences of images like this:

Since the speckles are mainly generated by interference effects on surface unevenness on the sample, the movement of the speckles on the surface can be measured by tracking user-definable areas.

A special algorithm based on a cross-correlation of two consecutive images determines the movement of the areas in the image.

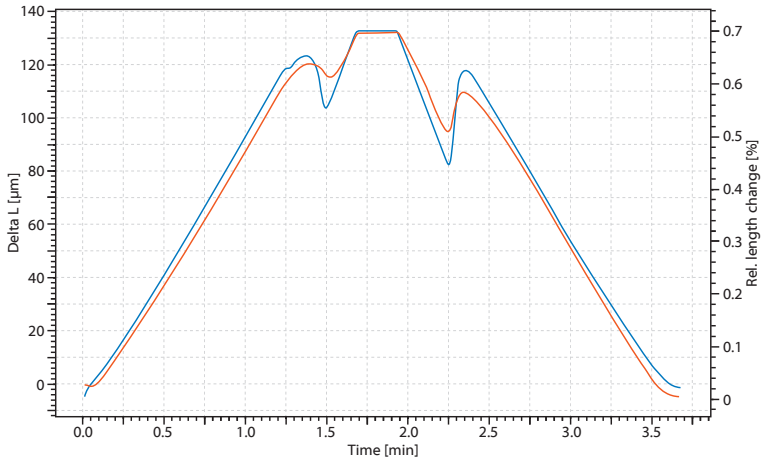
The size and position of the areas is user-definable. Additional to tracking the total distance of the areas the tracking direction can be constrained to vertical and horizontal direction.



This allows for measuring anisotropic expansion behavior of samples. The measured expansion is independent from pushrod expansion.

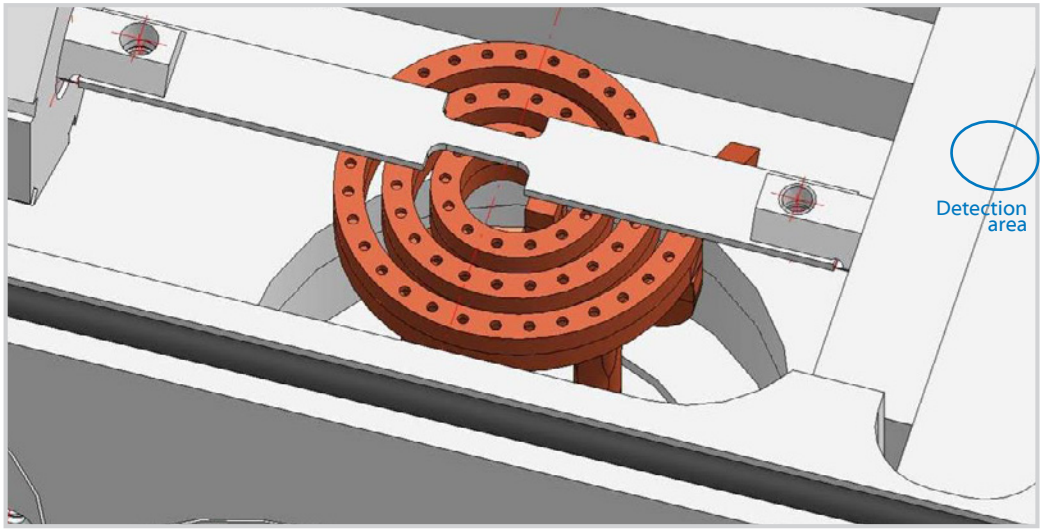
A big advantage compared to the LVDT measurement is, that the temperature over the small area recorded by the camera is distributed much more homogeneously than over the entire sample length. So microstructural transitions can be seen more clearly than with the LVDT.

Comparison to push rod measurement



This image shows the measuring signal of the LVDT in X-direction (black) and the measuring signal of the speckles-camera in Y-direction (blue). Start and finish points of phase transitions can be observed more clearly.

Tensile samples



For tensile samples it is a big advantage that the sample dilatation can be measured in both, X- and Y-direction. Measuring in Y-direction can give additional information about necking of the sample.

Real-time insight into the grain growth with the non-destructive NDT technology laser ultrasound system LUS

In cooperation between Linseis Messgeräte GmbH and RECENDT GmbH (Research Center for Non-Destructive Testing GmbH, www.recendt.at), a real-time grain size determination system based on a dilatometer system (DIL L78/RITA) and an adapted laser ultrasound system (LUS) was developed

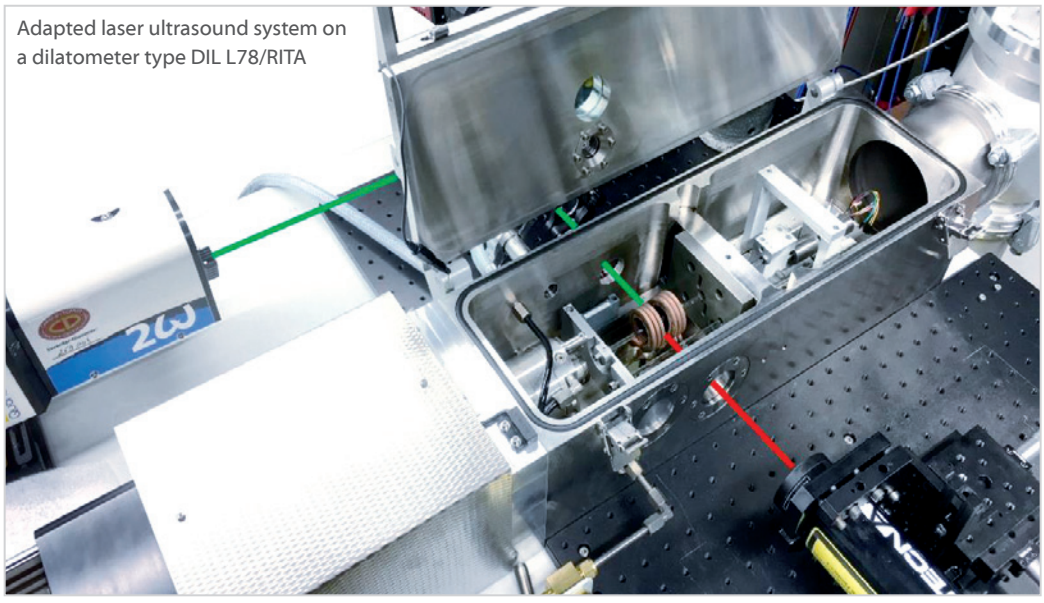
The grain size is determined from the LUS data as follows:

The non-destructive NDT technology "laser ultrasound" (LUS) enables an in-situ analysis of the grain size based on an evaluation of the frequency-dependent ultrasound attenuation $\alpha(f)$, which is mainly caused by the scattering at the grain boundaries due to the applied method.

The frequency-dependent ultrasound attenuation is modeled using the following power-law:

$$\alpha(f) = \alpha + bf^n$$

The attenuation coefficient $\alpha(f)$ is composed of an absorption coefficient *a*, a scattering coefficient *b*, the frequency *f* and the exponent *n*, where the absorption coefficient describes the internal friction losses and the scattering coefficient is the interesting grain size parameter (proportional to the mean grain size). The exponent n results from the ratio of the acoustic wavelength to the mean grain size, where three types of scattering can be distinguished, Rayleigh (*n*=4), Stochastic (*n*=2), and Geometric



Adapted laser ultrasound system on a dilatometer type DIL L78/RITA

Scattering[1].

The relationship between the scattering coefficient and the grain size of interest *D* is modeled as follows:

$$\alpha(f) = a + C\,(D - D0)^{n-1} f^{n}$$

The scattering coefficient *b* is the product of the material-dependent parameter *C* and the relative change in the mean grain size $D–D_0$ (D_0 - grain size of the initial state). A calibration of the model using mean grain size values from micrographs at certain temperature conditions provides the parameter *C* [2].

Laser ultrasound measurements and data analysis using this attenuation model provide real-time insight (in-situ) into the grain growth of a material during thermal cycles. Figure 2 shows an impressive comparison of these LUS real-time results (points) with several time-consuming micrograph analyzes (colored X markings).

[1] S. Sarkar, A. Moreau, M. Militzer, and W. J. Poole, "Evolution of austenite recrystallization and grain growth using laser ultrasonics," Metall. Mater. Trans. A Phys. Metall. Mater. Sci., vol. 39 A, no. 4, pp. 897–907, 2008, doi: 10.1007/s11661-007-9461-6.

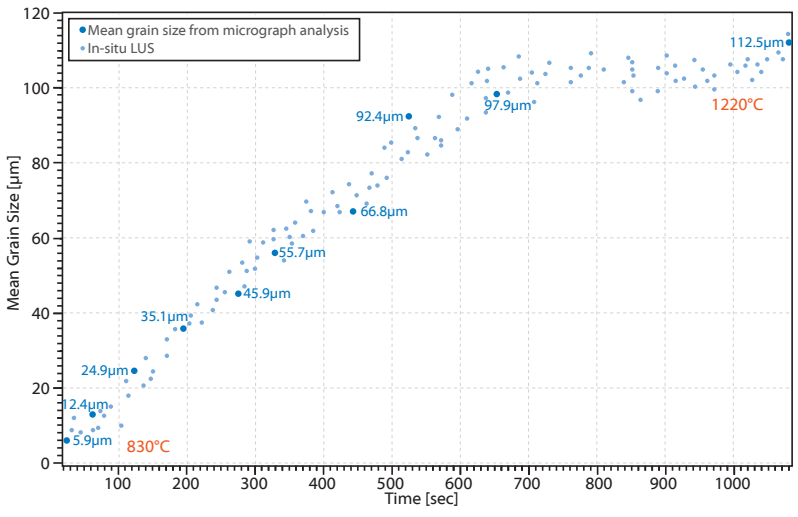
[2] T. Garcin, J. H. Schmitt, and M. Militzer, "In-situ laser ultrasonic grain size measurement in superalloy INCONEL 718," J. Alloys Compd., vol. 670, pp. 329–336, 2016, doi: 10.1016/j.jallcom.2016.01.222.

The result is very similar to a length change curve of a conventional quenching dilatometer, but the camera image is only about 1.6mm x 1.6mm large. So the transformation points are much sharper and even very weak transformations can be observed.

Additionally it is possible to calculate the absolute length change between two certain regions to get a result in µm.

Technical Specifications

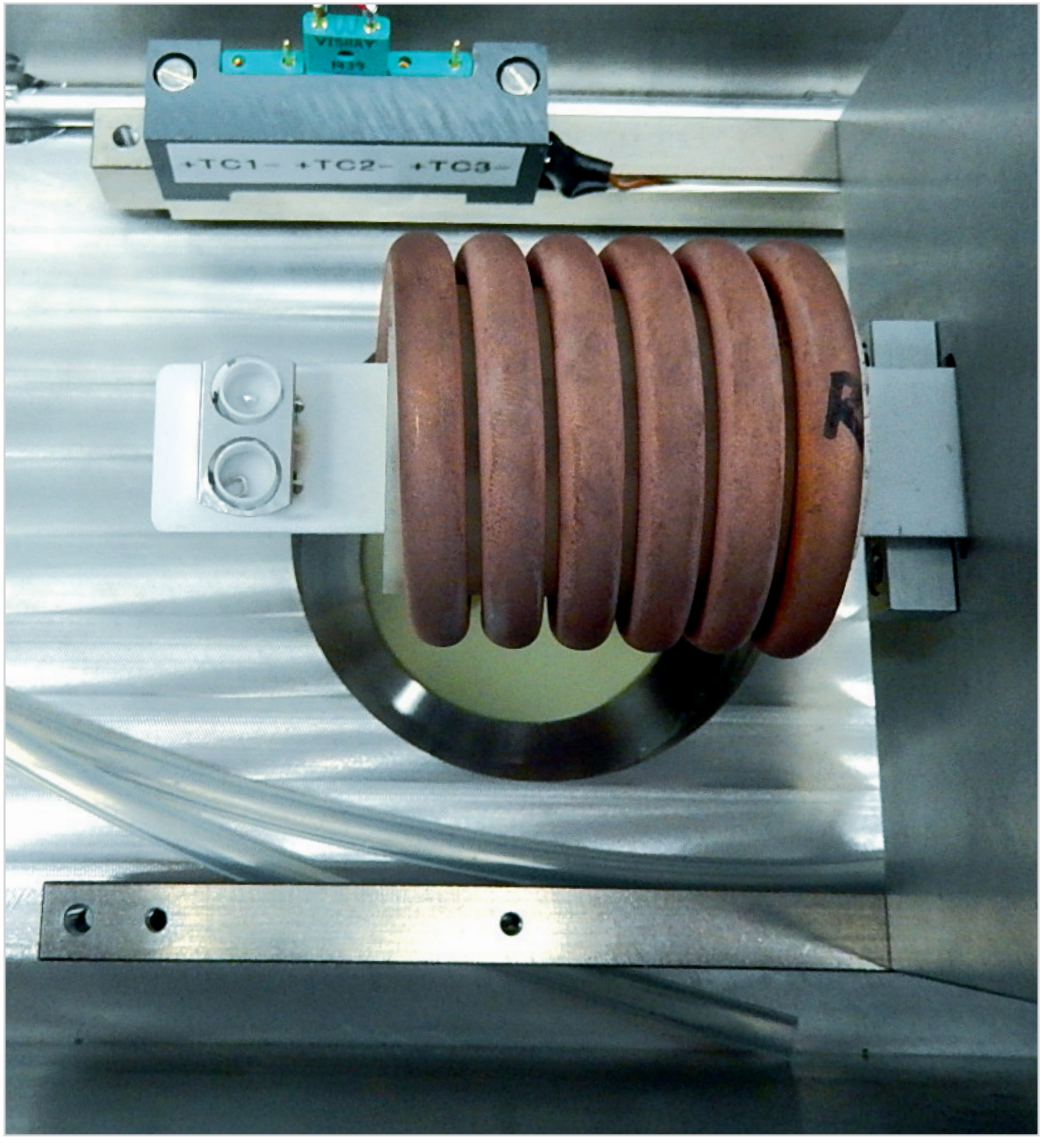
Resolution	1024x1024 px
Framerate	50 fps
Image size	1.6 x 1.6mm³ … 11 x 11mm²



DSC – DIFFERENTIAL SCANNING CALORIMETRY

Differential Scanning Calorimetry (DSC) is the most popular thermal analysis technique. It measures endothermic and exothermic transitions as a function of temperature.

- Endothermic = heat flows into a sample
- Exothermic = heat flows out of the sample



SOFTWARE

Features -Software

In respect to thermal and mechanical sample treatment numerous different mathematical functions can be selected.

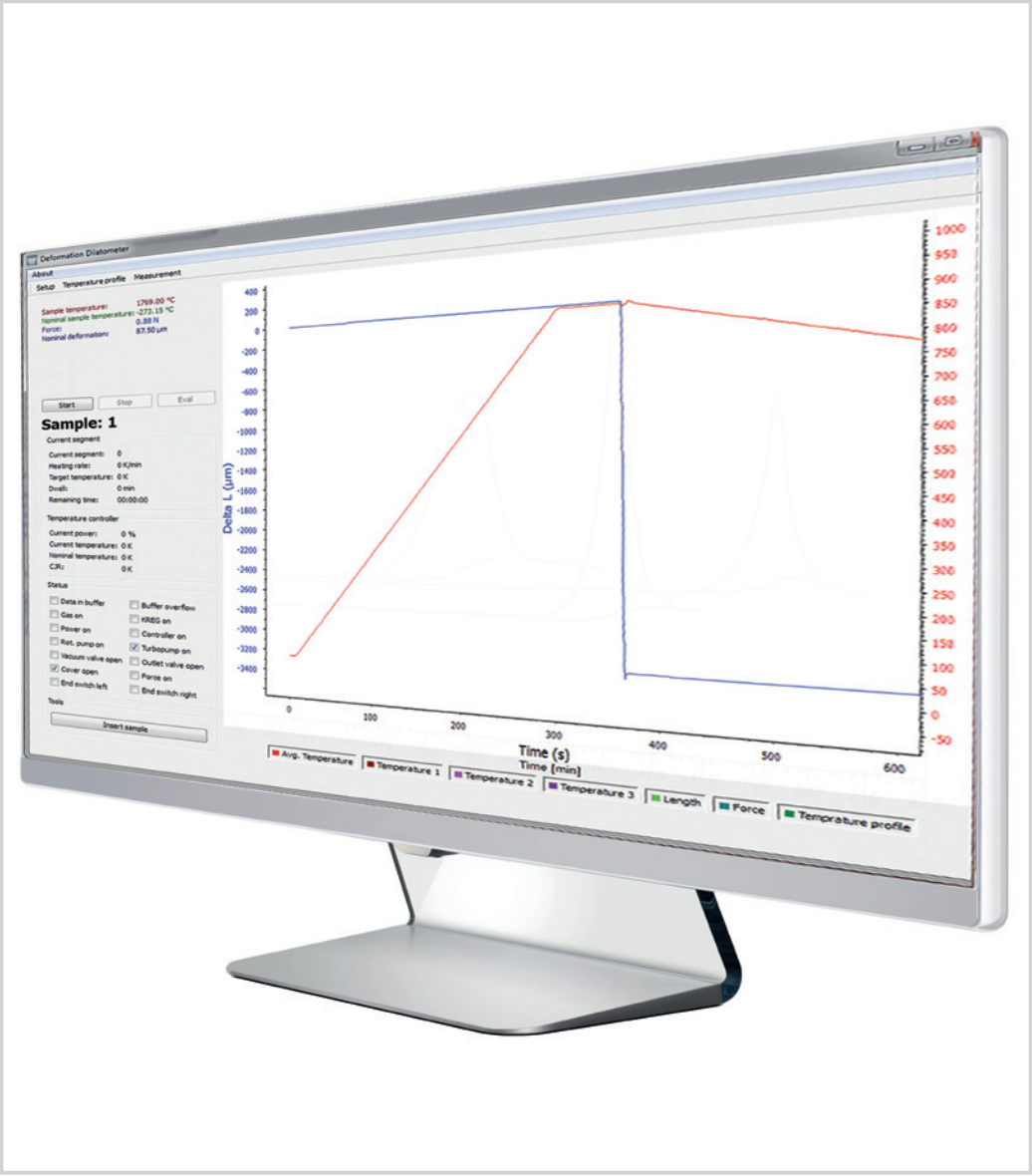
- Zoom function
- User-friendly
- Multi-methods analysis (DSC TG, TMA, DIL, etc.)
- Online help menu
- Report generator
- Data export to MS Excel
- Export and import of data ASCII
- Program capable of text editing
- Data security in case of power failure
- Thermocouple break protection
- Repetition measurements with minimum parameter input
- Evaluation of current measurement
- Storage and export of evaluations
- Programmable gas control
- Statistical evaluation package
- Smoothing of total or partial measurement
- Tangent intersection determination (automatic or manual)
- Free scaling

The information of a thermo analytical measurement can be increased when using the broad range of specialized Software.

Control and Evaluation Software

- Latest Windows operaing system
- Software for creation of CHT, CCT, DCCT and TTT diagrams
- all necessary measuring parameter are included in the menu structure
- Multiple forming steps during measurement
- Free choice of all control parameters
- Specification of temperature-time force-gradient and strain rate and deformation degree
- Control rate input by user or selection of industry parameter, i.e. quenching rates based on T 8/5 times
- Individual commentaries
- Heat up and cool down speeds
- End of the heating curve as well as duration of holding temperature
- Programmable heating / cooling and isothermal segments
- Function menus are easy to handle
- Graphical evaluation software with many functions to get complete results of all measured data
- Free assignment of axes
- The evaluation software includes freely scalable isothermal and continuous diagrams
- Manual entry of transition points
- Correction of individual data points
- Insertion of text
- ASCII Export

- Calculation of Delta L, Alpha physical, Alpha technical (CTE)
- Mathematical calculation of curves
- Statistical evaluation of curves with mean and confidence interval
- Print out of the results as curve or table
- Evaluation can be done simultaneously to an ongoing measurement / multi tasking
- Function menus are easy to handle
- Graphical evaluation software with many functions to get complete results of all measured data
- Free assignment of axes
- The evaluation software includes freely scala ble isothermal and continuous diagrams

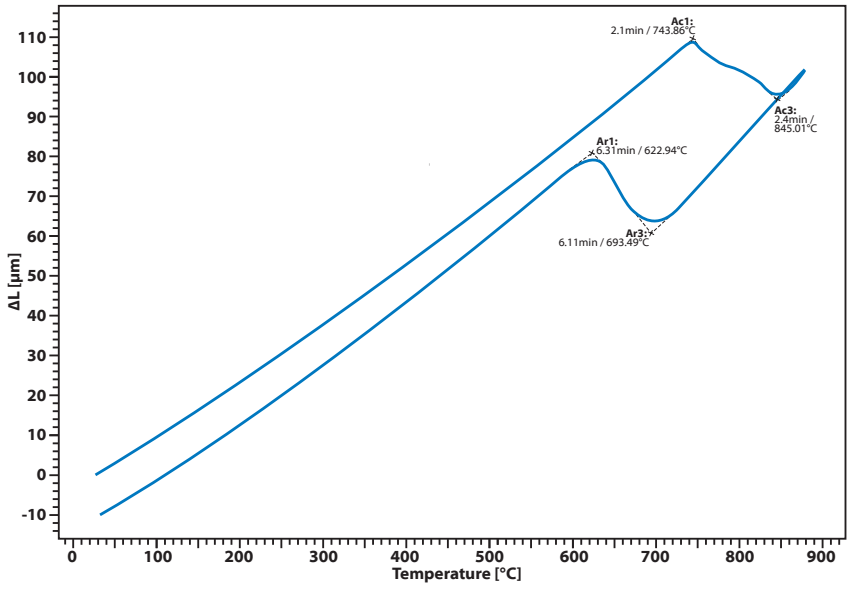


SPECIFICATIONS

	L 78 Q Stand alone Quenching Dilatometer	**L 78 RITA Q/D** Modular Quenching + Deformation + Tension	**L 78 RITA Q/D/T** Modular Quenching + Deformation + Tension
Temperature range	-150 up to 1600°C	RT up to 1600°C (RT up to 1700°C on request)	RT up to 1600°C (RT up to 1700°C on request)
Sample geometry	solid and hollow samples OD = 3 - 4 mm, L = 10 mm optional: OD = up to 12 mm optional: L = up to 60 mm	solid and hollow samples OD = 3 - 4 mm, L = 10 mm optional: OD = up to 12 mm optional: L = up to 60 mm	solid samples OD = 5 mm, L = 10 mm optional: OD = up to 12 mm optional: L = up to 60 mm
Heating principle	Induction heating	Induction heating	Induction heating
Heating rates	≤ 4000 K/s	≤ 125 K/s	≤ 125 K/s
Cooling rates	≤ 4000 K/s	≤ 125 K/s	≤ 125 K/s
Deformation force			up to 22 kN
Deformation rate			0.005 up to 100 mm/s (0.005 up to 200 mm/s on request)
True strain			0.02 to 1.2
Deformation			max. 7 mm
Resolution	5 nm	20 nm	20 nm
Minimum pause between two deformation steps			40 ms
Atmospheres			protective gases, vacuum down to 10^{-5}mbar
Mechanical control modes			stroke, force, rate, strain (optional)

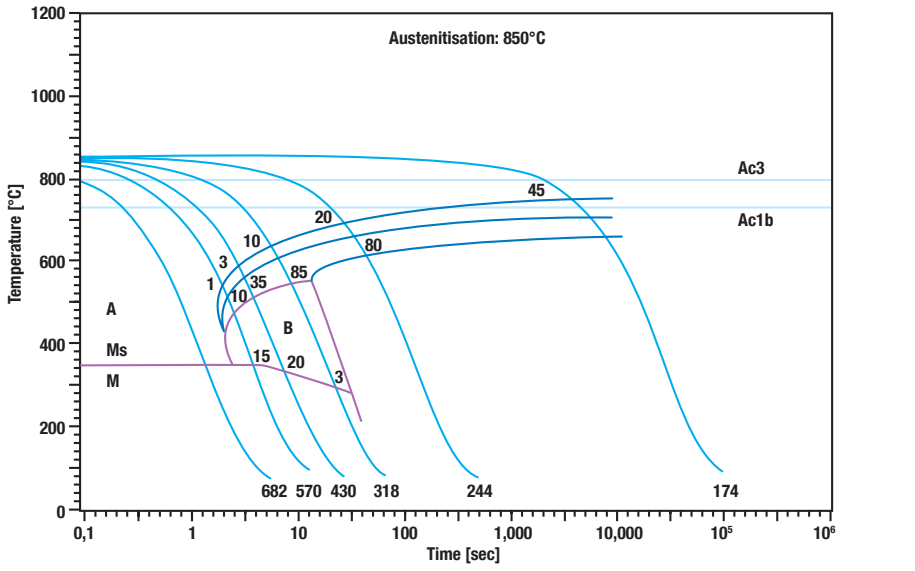
APPLICATIONS

Steel Phase Transformation



With the L78 Q and L78 Q/D Dilatometers the phase transformations in steel can be measured very accurately up to high heating and cooling rates. The transitions between different phases of steel are and the temperatures at which they occur are critical in the construction of the TTT, CCT and CHT diagrams. In this example the steel sample is heated in a first ramp above above its austenitic temperatur. Then the sample is quenched cooled. The plot shows the start (Ar3) and finish (Ar1) of the phase transformation from austenite to ferrite. These two temperature points can then be fitted to a CCT diagram based on the quench rate.

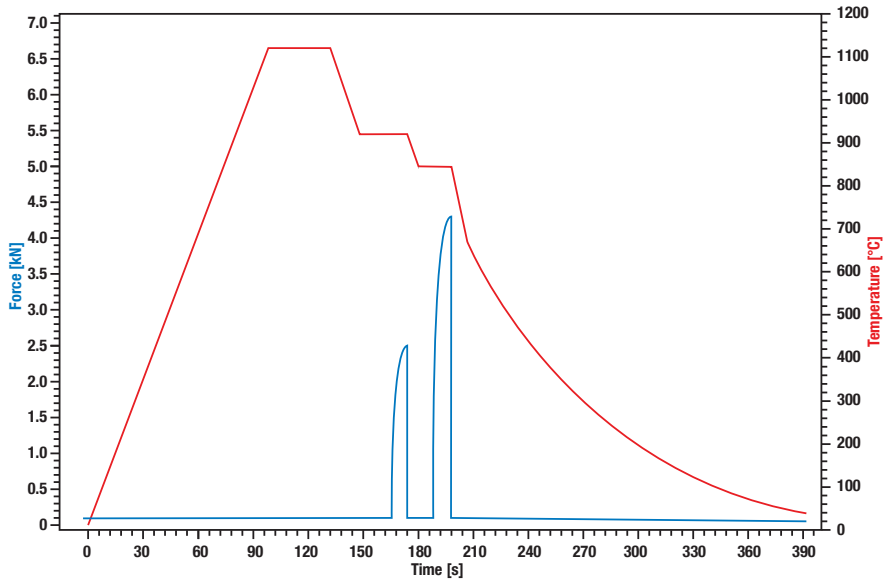
Continuous Cooling Transformation Diagram (CCT)



Picture © Dr. Sommer Werkstofftechnik GmbH, Issum

The CCT phase diagram represents the phase transformation of a material when it is cooled at various controlled rates. CCT diagram allow the prdeiction of the final microstructure of the measured steel. This crystalline structure determines the physical properties of the material. The L78 Q and L78 Q/D is the ideal tool to observe small dimensional changes under extreme conditions of controlled cooling. With the intuitive Software it is easy to prepare CCT, CHT and TTT diagrams from the test results.

2-Step Deformation Test



The L78 Q/D is the ideal instrument for optimizing the quench rate after multi-step deformations. With these measurements the processing of steel can be simulated to control the crystalline structure and the physical properties.
In this example, after the initial heating and resultant thermal expansion, the parcel of steel is held isothermally and goes through a series of 2 deformation steps: an initial 1mm deformation over a 10 s time period followed by a second 1 mm deformation over again 10 s time period. After the deformation steps the material is quenched and the contraction and phase transformation is measured. These data can be used for manufacturers to optimize their production processes for steels with the requested physical properties.

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