TA Instruments

DILATOMETRY

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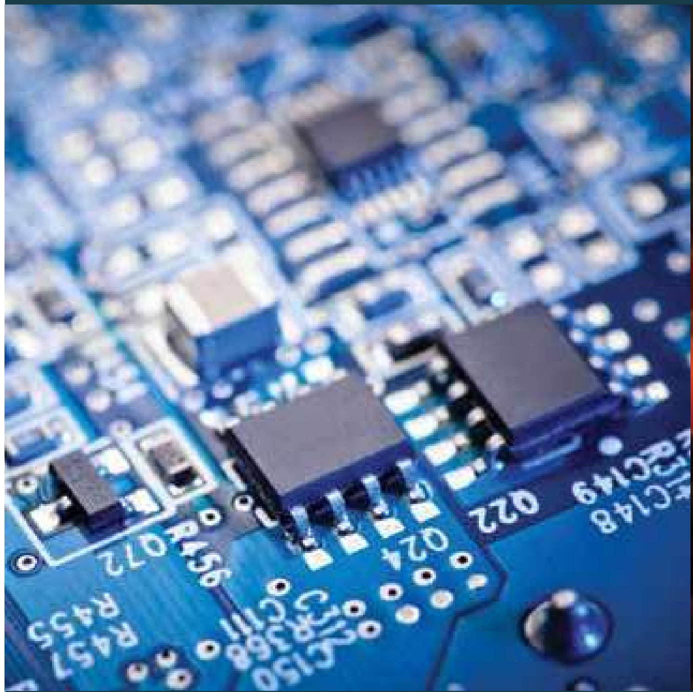
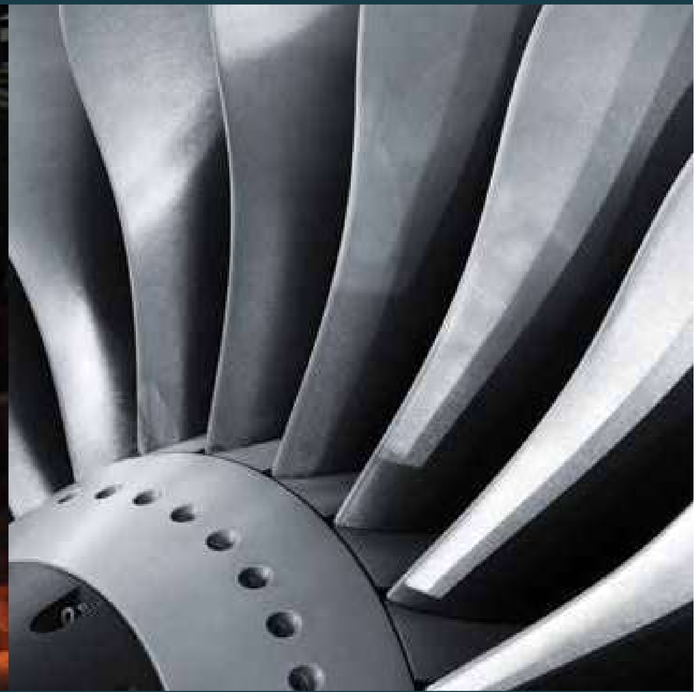




dilatometry

Every TA Instruments dilatometer precisely measures dimensional changes of a specimen brought about by changes in its thermal environment. Typical measurements include thermal expansion, annealing studies, determination of phase transitions and the glass transition, softening points, kinetics studies, construction of phase diagrams and sintering studies, including the determination of sintering temperature, sintering step and rate-controlled sintering. Investigation of processing parameters as reflected by dimensional changes of the material can be studied in great detail through exact duplication of thermal cycles and rates used in the actual process.

Each application of dilatometry has its own experimental requirements. That is why TA Instruments provides dilatometers in four basic types, each of which have flexibility of sample atmosphere, temperature and measurement control. Only TA Instruments can provide the right instrument to match your needs—no matter what your application may be.

dilatometer

DIL 805

In the heat treatment of metal alloys, the heating rate, quenching rate and isothermal dwell times are important parameters that dictate the final crystalline structure and the resultant physical properties. These microstructural changes may be observed through process simulation with real-time monitoring of dimensional change. Among other things, measurements of distinct alloy compositions are used to create time-temperature-transformation diagrams (TTT) and continuous-cooling-transformation diagrams (CCT), which are critical in process design and optimization. The DIL 805 series quenching dilatometers provide the most accurate measurements over the widest range of heating, cooling and deformation conditions, allowing for the most sophisticated characterization and optimization of metals processing.

DIL 805L

DIL 805D

	DIL 805L	DIL 805A	DIL 805D
Temperature Range (dependent on sample material)	20 °C to1500 °C -150 °C to 1300 °C	20 °C to 1500 °C -150 °C to 1300 °C	20 °C to 1500 °C
Heating Principle	Inductive	Inductive	Inductive
Heating Rate	≤ 2000 K/s	≤ 4000 K/s	100 K/s
Cooling Rate	≤ 2500 K/s	≤ 2500 K/s	≤ 100 K/s
Sample Material and Geometry	electro-conductive solid or hollow samples OD=4 mm, L=10 mm		electro-conductive solid samples OD=5 mm, L=10 mm
Atmosphere	air, vacuum, inert gas		air, vacuum, inert gas
Resolution (ΔL/°C)	0.05 µm / 0.05 °C		0.05 µm / 0.05 °C
Deformation Force			≤ 20 kN
Deformation Rate			0.01 mm/s to 200 mm/s
Strain Rate $\dot{\varphi}$			0.001 to 20.0 s^{-1}
True Strain φ			0.05 - 1.2
Deformation			max. 7 mm
Number of deformation steps			Unlimited
Min. pause between deformation steps			40 ms

quenching dilatometer

DIL 805 ACCESSORIES

805L Quenching Dilatometer

The DIL 805L is a fully automated self-contained quenching dilatometer used to observe dimensional changes under extreme conditions of controlled heating and cooling. A solid or hollow sample is inductively heated to a temperature plateau and is then continuously cooled at a user-defined (linear or exponential) cooling rate. The phase transformation occurring in the continuous cooling process or in the isothermal dwell (which may also be a multistep transition) is indicated by the measured change in length. An array of cooling or isothermal curves represents a continuous-cooling-transformation (CCT) diagram or an isothermal time-temperature-transformation (TTT) diagram, respectively. The beginning and the end of the transformation indicate the alloy phase boundaries, e.g. ferrite, carbide, graphite, pearlite, bainite, martensite, or other eutectoid phase batches. Test experiments are flexible and may be constructed to mimic a process of any length or complexity.

805A Quenching Dilatometer

The 805A Quenching Dilatometer is the new benchmark technology for determining the dimensional changes and phase transformations of steel alloys that require the most stringent of temperature controls. Operating from -160 ˚C to 1500 ˚C (2 temperature configurations) with heating rates of up to 4000 K/s and cooling rates in excess of 2500 K/s, tests can be conducted to closely simulate the material response for any production or heat treatment process. It is designed to accommodate many different add-on modules, including the 805D Deformation adaptor, 805T Tension adaptor, and the DTA/DSC measuring head. This instrument is a powerful and versatile tool for the determination of critical parameters in steel manufacturing and heat treatment processes.

Sub-zero Module

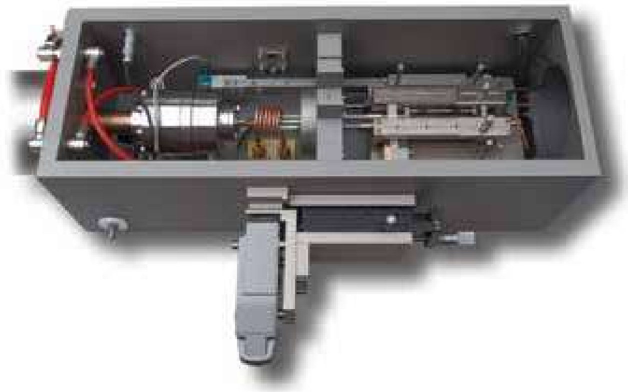
In many cases the martensitic finish temperature, Mf, of a steel lies well below room temperature. This add-on module operates from -160 ˚C to 1300 ˚C, with attainable controlled quenching rates in excess of 2500 K/s, and allows for the complete characterization of the austenite to martensite transformation. This unique quenching technology passes helium gas through a copper heat exchanger submersed in a liquid nitrogen bath before delivery to a hollow sample. The design greatly improves heat transfer by eliminating many of the issues associated with liquid nitrogen cooling, including condensation, material interaction, and imprecise response rates.

805D Deformation Dilatometer

Steel processes, such as hot or cold rolling, require detailed knowledge of the time-temperature-transformation diagram after deformation (DTTT diagram). With the deformation module, the principle of the 805A quenching dilatometer is extended to include controlled deformation. Solid samples are compressed using various deformation programs (e.g. linear, multi-level with a constant deformation force or rate) with controlled forces up to 25 kN or rates up to 200 mm/s. An unlimited number of deformation steps can be performed with a pause between steps of only 40 ms. This unique technology enables the control of cooling and deformation processes in order to create a DTTT diagram. The 805D is also used to examine creep and relaxation processes.

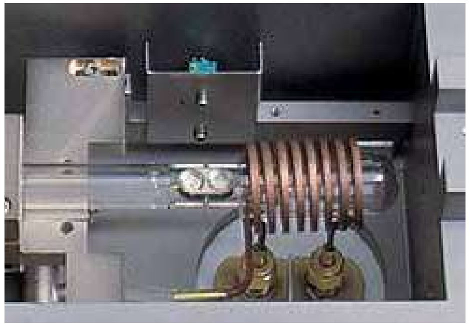
805T Tension and Compression Adapter

The 805T extension further extends the capabilities of the instrument to alternating tensile and compressive loading. The expansion of a clamped sample is measured during heating or cooling to emulate mill processing. Once the desired temperature is achieved, it is held isothermally while the desired mechanical cycling is performed. Force-controlled or strain-controlled cycles are available up to 8 kN or 20 mm/s, respectively. Additionally, tensile loading to fracture lends additional information about the final performance characteristics of the material. These data are used to generate true-stress vs. true-strain or stress/strain cycling plots.



Alpha Measuring System

The Alpha measuring head uses low-expansion fused silica components in conjunction with a true differential LVDT for high-precision expansion measurements. This system allows the DIL 805 to be used for traditional push-rod dilatometer studies such as the determination of the coefficient of thermal expansion (CTE) and the softening point.

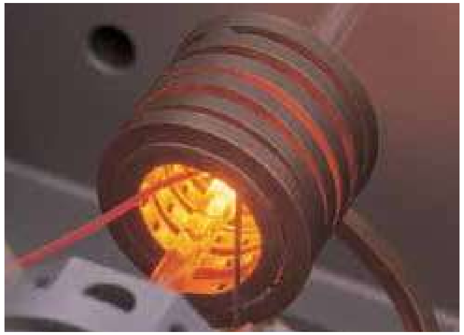


DTA/DSC Measuring Head

The DTA/DSC measuring head is custom-designed for the analysis of phase transformation and precipitation processes in metals. With heating and cooling rates of up to 500 K/min, an identical temperature program (as in many of the quenching studies) can be used in conjunction with a traditional thermal analysis measurement.

Induction Heating Coil

The custom-designed induction heating coil allows for rapid inductive heating at rates up to 4000 K/s of an electrically conductive solid or hollow sample. During a test, only the sample is heated so there is no associated furnace/insulation cool-down period, and another sample can be loaded immediately upon test completion. The hollow-core inner coil also serves as the purge gas conduit focused at the heating zone, ensuring an inert environment throughout the test. Specially designed heating rings are also available for use with samples that are not electrically conductive.



Optical Module

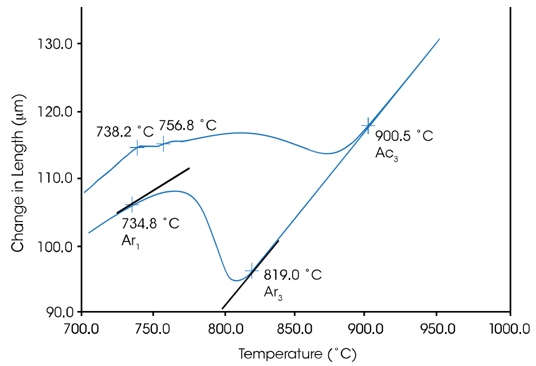
Traditional dilatometers measure the thermal expansion of a material in one axial direction and have an inherent drift associated with the thermal interaction at the contact point between the push-rod and the sample, especially during isothermal dwells. With the optical expansion module, contraction/expansion is monitored in two directions during the test run. The measurement is non-contact and absolute, so it is free from interaction with dilatometer temperature gradients and a calibration correction is not necessary. The optical module is available for the quenching, deformation and tension/compression configurations, and produces results that are unachievable by conventional methods.

Thermocouple Placement Device

Precise temperature control requires temperature monitoring in close proximity to the sample. The easy-to-use thermocouple placement device reproducibly spot welds up to 3 thermocouples directly onto the sample for temperature resolutions of 0.05 °C across the full temperature range. The welding current and time, the contact pressure, and the inert gas purge can be adjusted to ensure a strong spot weld onto the sample.

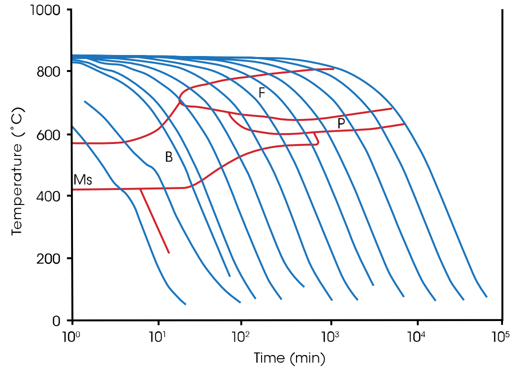
Steel Phase Transformation

Phase transformations in steel are highly path dependent, reflecting the effects of earlier processing steps on subsequent phase composition. The transitions between different phases of steel are especially clear when measured by the DIL 805A Quenching Dilatometer, and the temperatures at which they occur are critical in the construction of the TTT and CCT diagrams. In this example, the first ramp rate heats the sample above its austenitic temperature, at which time it is quenched. The plot shows the start (Ar_3) and finish (Ar_1) 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.



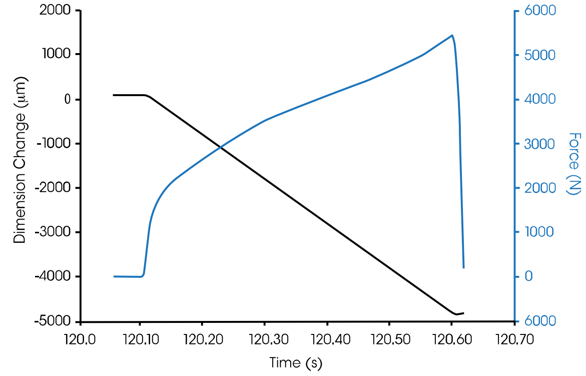
Continuous Cooling Transformation Diagram

As the name suggests, the CCT phase diagram represents the phase transformation of a material when it is cooled at various controlled rates. In the heat treatment of steel, the CCT diagram is used to predict the final crystalline structure of the processed steel. This crystalline structure determines the physical properties and suitabilty for the application in which the material will be used. The DIL 805A is the ideal tool to observe small dimensional changes under extreme conditions of controlled cooling. Software is available for the seamless preparation of TTT or CCT diagrams.



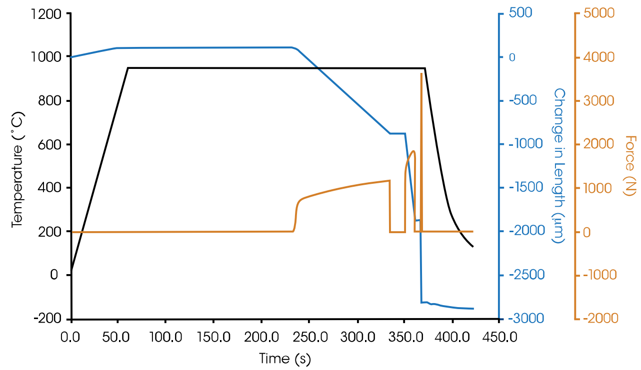
Force Deformation

The 805D add-on module can precisely control the strain-rate of a sample and measure the resultant force required to achieve this. In this high speed test run, a deformation rate of 10 mm/s is used for a maximum displacement of 5 mm (Strain 0.50). The force exerted by the hydraulic ram is closely monitored, and both data sets can be used to plot the true stress vs. true strain curve of the material.



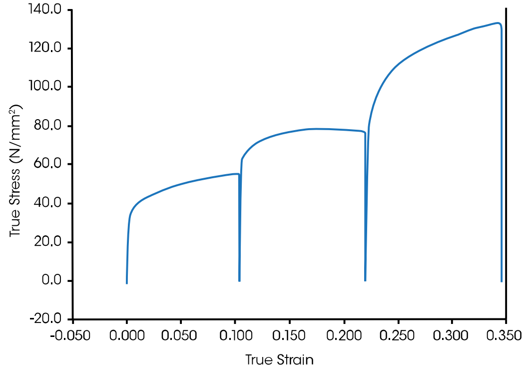
3-Step Deformation Test

Simulating metal processing techniques, and the phase transformations that take place upon quenching or heat treating, are important measurements to perform in order to accurately control the crystalline structure and its inherent physical properties. The DIL 805A/D is the ideal instrument for optimizing the quench rate after these multi-step deformations. In this example, after the initial heating and resultant thermal expansion, the parcel of steel is held isothermally and goes through a series of 3 deformation steps: an initial 1mm deformation over a 100 s time period; a second 1 mm deformation over a 10 s time period, and finally a seemingly instantaneous force applied for the final 1 mm deformation. After another 10 s dwell at the isothermal processing temperature, the material is quenched and the contraction and phase transformation is measured. Using this measured data, the manufacturer can streamline their processing for repeatable production of steel with the desired physical properties.



True Stress vs. True Strain Curves

This plot is the true stress vs. true strain curves, measured during the deformation steps in the above example. Please note that the "instantaneous" force pulse in the third deformation step was measured and can now be analyzed. With over 100,000 data points taken per second, the DIL 805A/D is a powerful tool that can further help engineers develop the mechanical aspects of the processing line.



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