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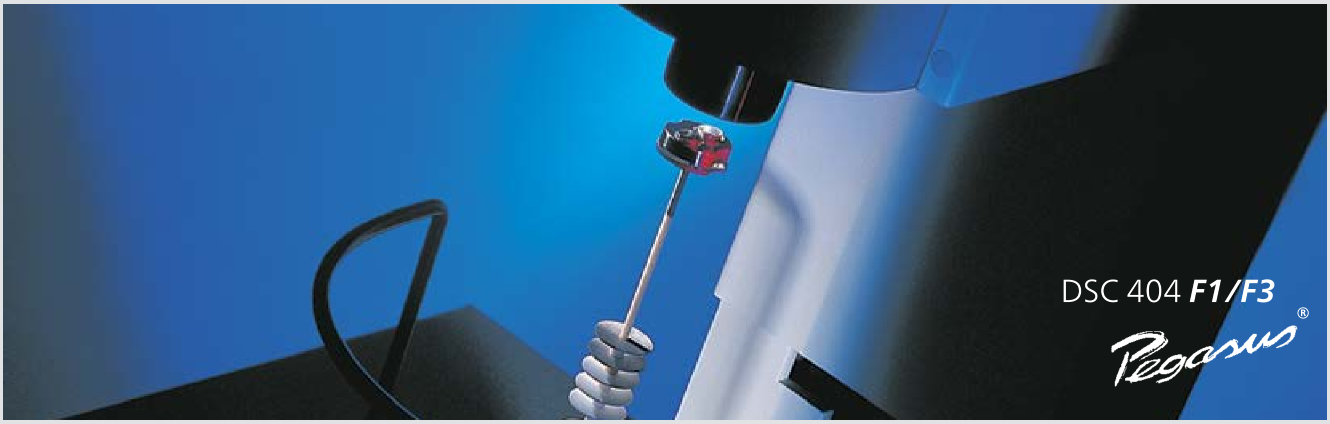
Analyzing & Testing

Differential Scanning Calorimetry

Method, Technique, Applications



Leading Thermal Analysis

DSC 404 ***F1/F3*** *Pegasus*® – Method

Differential Scanning Calorimetry

Differential scanning calorimetry (DSC) is one of the most frequently employed thermal analysis methods. It can be used to analyze nearly any energetic effect occurring in a solid or liquid during thermal treatment.

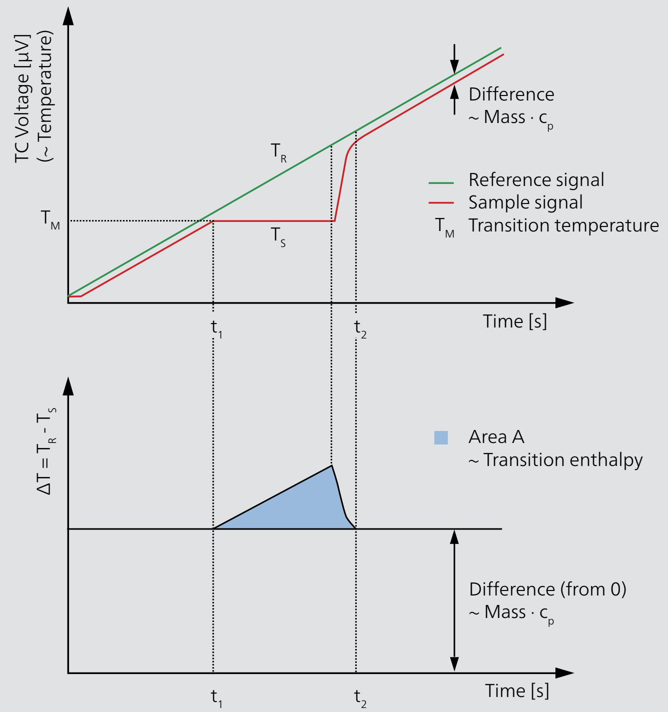
DSC Analysis Possibilities

- Specific heat capacity
- Melting and crystallization behavior
- Solid-solid transitions
- Polymorphism
- Phase transitions/diagrams
- Liquid crystal transitions
- Eutectic purity
- Degree of crystallinity
- Glass transition temperatures
- Cross-linking reactions
- Oxidative stability
- Decomposition onset
- Compatibility
- *Purity Determination*
- *Thermokinetics*

DSC Method

The DSC 404 ***F1*** and ***F3*** *Pegasus*® systems operate according to the heat flux principle. With this method, a sample and a reference are subjected to a controlled temperature program (heating, cooling or isothermal). The actual measured properties are the temperature of the sample and the temperature difference between sample and reference. From the raw data signals, the heat flow difference between sample and reference can be determined.

The DSC 404 ***F1/F3*** *Pegasus*® instruments meet nearly every respective instrument and application standard, including:
ISO 11357, ASTM E967, ASTM E968, ASTM E793, ASTM D3895, ASTM D3417, ASTM D3418, DIN 51004, DIN 51007, DIN 53765.



Signal generation in a heat flux DSC

DSC 404 ***F1/F3*** *Pegasus*® – Outstanding Features

Pure Flexibility – Furnace Variety for the Broadest of Temperature Ranges

Various furnaces including those for special applications extend the application range beyond that of common thermal analyzer systems. Measurements can be performed in the range from -150°C to 2000°C.

Double Hoist — High Sample Throughput

The double hoist allows for either the simultaneous connection of two furnaces or one furnace in combination with the automatic sample changer (ASC).

Vacuum-Tight Design for Elimination of Atmospheric Influences

Mass flow controllers provide an ideal means of testing materials sensitive to oxidation. Enthalpy changes and the specific heat (c_p) can be analyzed at unmatched levels of accuracy.

Pure Flexibility – Sensor Variety

The instrument's ample flexibility is extended by an impressive assortment of sensors. The great variety of possible furnace/sensor combinations ensure an optimal configuration for any application. Easy handling is guaranteed by means of the user-friendly design.

Efficient Time Management

The automatic sample changer (ASC) is designed to handle up to 20 samples. Flexibility and high sample throughput allow for efficient time management.

Pure Flexibility – Acessories for Special Configurations

A variety of optional accessories and special configurations are available to ideally optimize the base system and adjust it to specific needs.

Pure Flexibility in Groundbreaking Technology

Vacuum-Tight Design – Optimum Atmosphere Control

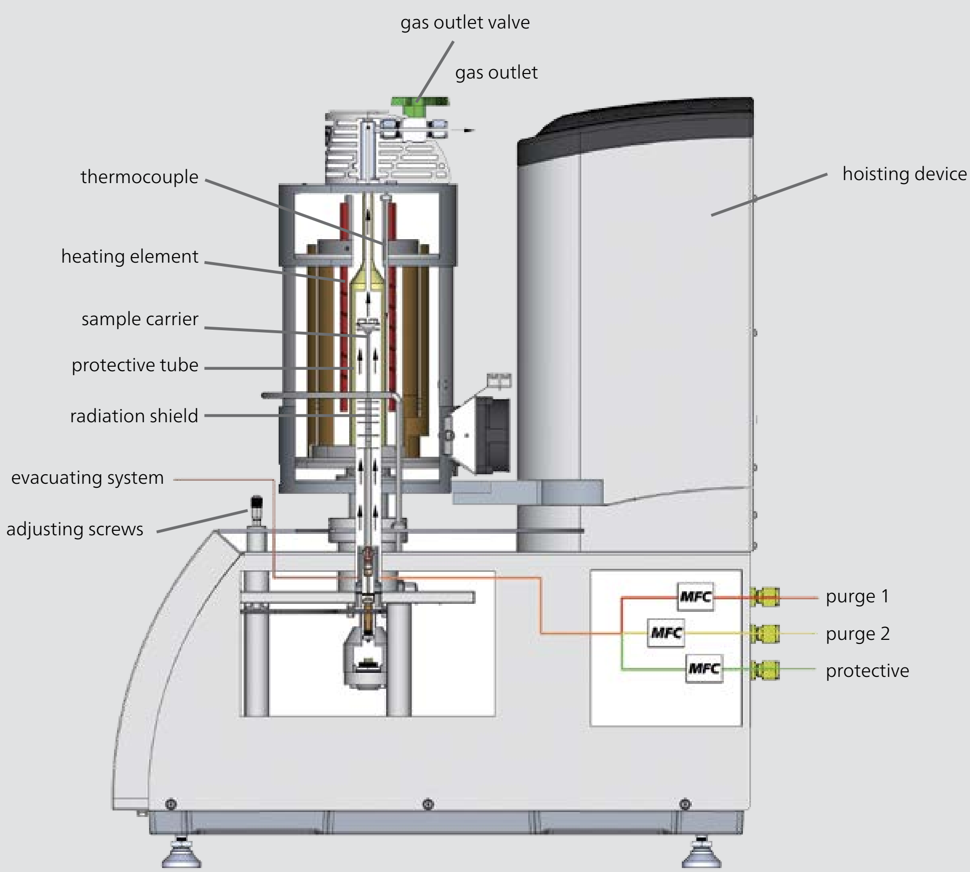
The DSC 404 ***F1/F3*** *Pegasus®* instruments stand out as first-rate vacuum-tight high-temperature DSCs. Practically every component is constructed to fulfill the requirements of high-vacuum and high-purity gas applications. Various pump systems are available to evacuate the system down to 10^{-4} mbar. Automatic evacuation is of course possible, as is backfilling with various kinds of purge gases.

The *OTS®* accessory can be used to drastically reduce the oxygen concentration at the sample to below 1 ppm.

Atmosphere – Mass Flow Controllers

In the DSC 404 ***F3*** *Pegasus®*, the purge or reactive gas flow is generally controlled via frits, manual control systems or tailor-made mass flow control systems (MFCs). The DSC 404 ***F1*** *Pegasus®* comes standard-equipped with integrated metal-housed mass flow control systems for three different gases.

Both MFC systems allow software-controlled gas switching and purge gas rates as well as recording of the flow rates in the software.



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Various Furnace Systems

The DSC 404 ***F1/F3*** *Pegasus*® systems can be equipped with a wide range of different furnaces accommodating different temperature and application ranges between -150°C and 2000°C. Silver and steel furnaces are available for the sub-ambient temperature range. Controlled cooling is guaranteed with the liquid nitrogen cooling device or the Vortex tube. For higher temperature ranges, SiC, Pt, Rh and graphite furnaces are available. For working under humid atmospheres in combination with the water-vapor furnace, a humidity generator is available.

Double Hoist

The electrically driven double furnace hoist is a standard feature of the DSC 404 ***F1/F3*** *Pegasus*® systems. It allows for simultaneous installation of two different furnaces to conduct, for example, low- and high-temperature tests with the same instrument. For an improved sample throughput, it is also possible to connect a single furnace along with the automatic sample changer instead.

Interchangeable Furnaces

Type	Temperature Range	Cooling System	Atmospheres
Steel	-150°C to 1000°C	Liquid nitrogen/Vortex	Inert, oxidizing, reducing, vacuum, corrosive
Silver[1]	-120°C to 675°C	Liquid nitrogen/Vortex	Inert, oxidizing, reducing, vacuum, corrosive
Water-vapor	RT to 1250°C[2]	Air	Up to 100% humidity
Silicon carbide	RT to 1550°C	Air	Inert, oxidizing, reducing, vacuum, corrosive
Platinum[1]	RT to 1500°C	Air	Inert, oxidizing, reducing, vacuum
Rhodium[1]	RT to 1650°C	Air	Inert, oxidizing, reducing, vacuum
Graphite	RT to 2000°C	Tap or chilled water[3] (or cooling thermostat)	Inert, oxidizing (with protective tube up to 1750°C), reducing

1 Optimally suited for c_p determination
2 Depending on the humidity content
3 Requires connection to cooling water

Pure Flexibility in Groundbreaking Technology

Various Sensors

The DSC 404 ***F1/F3*** *Pegasus®* instruments are generally used to obtain accurate specific heat (c_p) measurements. However, the systems allow for simple DTA measurements or conventional DSC tests as well. DTA sensors can be used for applications such as routine tests on aggressive sample substances. Various thermocouple types allow optimum sensitivity and time constants in all temperature ranges. The sensors can easily be changed in less than a minute by the operator.

Unique Sensor Adjustment System

For optimizing the baseline, a micrometer adjustment system is integrated into the measurement part. This adjustment system allows placement of the sensor at the optimum central position in the furnace. This guarantees a stable and reproducible baseline without any major adjustment efforts.

DTA sensor with protected thermocouples

DSC sensor in round design

DSC sensor

DSC sensor in octagonal design for ASC operation

Interchangeable Sensors

Thermocouple	Temperature Range	Sensor Types			Atmospheres				
		DTA	DSC	DSC-c_p	Inert	Oxidizing	Reducing[1]	Vacuum	Corros.
E	-150°C to 700°C	✓	✓	✓	✓	✓[4]	✓	✓	
K	-160°C to 800°C	✓	✓	✓	✓	✓[4]	✓	✓	
P	-150°C to 1000°C		✓	✓	✓	✓	✓	✓	
S	RT to 1650°C	✓	✓	✓[2]	✓	✓	✓	✓	
$S_{Protected}$	RT to 1650°C	✓			✓	✓	✓	✓	✓
B	RT to 1750°C	✓	✓	✓[3]	✓		✓	✓	
W/Re	RT to 2000°C	✓			✓		✓	✓	

[1] Upper temperature limit may deviate from the maximum temperature range of the sensor
[2] Optimum accuracy to 1500°C
[3] > 300°C to max. 1200°C
[4] Up to 500°C

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Technical Key Data of the DSC 404 *F1/F3 Pegasus*®

	DSC 404 ***F1*** *Pegasus*®	DSC 404 ***F3*** *Pegasus*®
Temperature range	-150°C to 2000°C	
Furnaces	Standard and special furnaces	
Heating/cooling rate	0.001 K/min to 50 K/min (depending on furnace type)	
Furnace hoist	Motorized double hoist for two furnaces or one furnace combined with the ASC	
Sensor types	DTA, DSC, DSC-cp, quick and safe interchange	
c_p measuring range[1]	Up to 5 J/(g*K)	
c_p determination[1]	Yes	Optional
TM-DSC	Yes	No
BeFlat® (DSC)	Yes	Optional
Tau-R® *Mode*	Yes	Optional
Purity Determination	Optional	Optional
Macro recorder for analysis routines	Yes	Optional
Gas atmospheres	Inert, oxidizing, reducing, vacuum (10^{-4} mbar)	
Vacuum-tightness	10^{-4} mbar (10^{-2} Pa)	
Gas control	3 integrated mass flow controllers (MFC)	3 integrated frits optionally 3 mass flow controllers (MFC)
Automatic sample changer (ASC)	20 samples, removable carousel	
Automatic evacuation	Software-controlled	Optional
Coupling to evolved gas analyzers	FT-IR, MS, FT-IR-MS, GC-MS, GC-MS-FT-IR	
*Pulse*TA®	Optional	

[1] c_p, specific heat capacity

A Diverse Array of Accessories

Plethora of Crucibles

What sets the DSC 404 ***F1/F3*** *Pegasus®* systems apart is not only their flexibility in furnaces and sensors but also the huge variety of available crucibles. For the broad temperature range from -150°C to 2000°C, the crucible materials vary from metals (Al, Ag, Au, etc.) to ceramics (Al_2O_3, MgO, ZrO_2, Y_2O_3, BN, etc.) to graphite. For inhomogeneous samples and those with low density, larger crucibles are available.

Should samples need to be shut off from the influence of the ambient atmosphere, or should gas emissions from the samples need to be contained, aluminum crucibles can be sealed shut, gas-tight, with a handy sealing press. For measurements under increased pressures of up to 100 bar, reusable stainless steel and titanium autoclave crucibles handle the job. A PtRh/ceramic crucible system for measurements on metal melts or other reactive test materials is available with a removable liner. Liners are available made of thin-walled Al_2O_3, MgO, and Y_2O_3.

Automatic Sample Changer (ASC)

An automatic sample changer (ASC) can be installed on both the DSC 404 ***F1*** and ***F3*** *Pegasus®* systems. The sample carousel allows for 20 samples and is removable for easy loading. Each sample can be assigned an individual measurement and evaluation macro. Preprogramming permits measurements to be carried out overnight or on weekends. This significantly enhances the sample throughput, laboratory and instrument efficiency.

Optimum crucible placement is guaranteed by design. The ASC with its gripper and carousel allows for the use of nearly any crucible type including specialties such as low-, medium- and high-pressure crucibles.

"Remove Lid" Function of the ASC

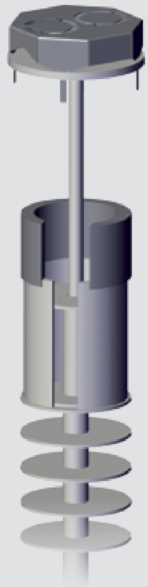
For unstable samples – i.e., samples sensitive to oxygen or ambient room conditions while waiting their turn on the crucible magazine to be inserted into the sample compartment – a "remove lid" device can be ordered with the instrument. Closing the crucibles with a lid minimizes the risk that critical samples would evaporate or react with ambient humidity prior to the measurement.

Reshaping tool for PtRh/ceramic crucbile system


Selection of DSC crucibles

AutoVac, _OTS®_, Evacuation Systems – Glove Box and Corrosion-Resistant Instrument Versions

A variety of useful accessories for augmenting the application range of the DSC method are available, including evacuation devices (from rotary or diaphragm to turbo molecular pumps), the *AutoVac* for automatic evacuation and refilling of the DSC system, and the *OTS®* oxygen trap system. In addition, special DSC versions are available for use in a glove box and for measurements in corrosive atmospheres. This great flexibility allows the DSC 404 ***F1/F3*** *Pegasus®* systems to be optimized and configured according to your needs – both now and in the future.



DSC Sensor – the *OTS®* device removes traces of residual oxygen in the gas atmosphere inside the instrument

Calibration Materials

For the calibration of temperature, enthalpy and specific heat in both the low- and high-temperature range, multiple sets containing different calibration materials are available for the various crucible materials (including high-pressure autoclaves).

The calibration materials are selected and prepared for measurement in accordance with the recommendations of the corresponding ASTM and CEI-IEC standards.

Proteus® Software – Key Features

Both DSC systems run under the *Proteus*® software on Windows® XP Professional, or on Windows® 7 32-/64-bit Professional, Enterprise or Ultimate. The *Proteus*® software includes everything you need to carry out a measurement and evaluate the resulting data. User-friendly menus combined with automated routines make this very easy to use while still providing sophisticated analysis. The *Proteus*® software is licensed with the instrument and can of course be installed on other computer systems.

Key Features of the General Software

- Mulit-tasking: simultaneous measurement and evaluation
- Multi-modeling: operation of different instruments with one computer
- Automatic detection of instrument settings (e.g., furnace, sensor, etc.)
- Storage of the analysis results and status for later restoration and continuation with analysis (original file remains)
- Results by e-mail
- Gas manager and gas control segment by segment
- Context-sensitive help system
- Presentation of DSC curve in absolute (µV, mW) or relative (µV/mg, mW/mg) units
- Calibration and correction routines for temperature, sensitivity, baseline
- Export of graphics with evaluation results to clipboard or to common formats such as EMF, PNG, BMP, JPG, TIF or PDF
- Data export in Excel®-compatible CSV-format
- ASCII file export of the raw data and/or the corrected measurement data for further data processing
- Multiple-window technique for clear presentation and evaluation of measurement curves or graphical excerpts in multiple windows
- Formatting of the results, measurement values, and axis labels optionally in technical format or as scientific notation
- Application languages: English, German, French, Russian, Chinese
- Print languages: English, German, French, Russian, Chinese, Italian, Spanish, Portuguese, Polish
- Software produced by ISO-certified company

Key Features of the Measurement Software

- Up to 256 programmable temperature segments
- Repetitive measurements with minimum parameter input
- Monitoring of all MFC gas flows
- Loop programming: insertion, deletion, and annexation of temperature segments, even in already existing temperature programs
- Snapshot for online evaluation of the measurement in progress
- Macro recorder for fully automatic analysis
- Special functions:
  - Jump to the next segment
  - Changing of upcoming segments during measurement
  - OIT (software-controlled optimization of measuring time: reset or jump to cooling segment)
- TM-DSC (only DSC 404 ***F1*** *Pegasus*®)
- Instrument Cockpit App: information on the current measurement status, direct access to the running test, ability to stop the measurement remotely anytime

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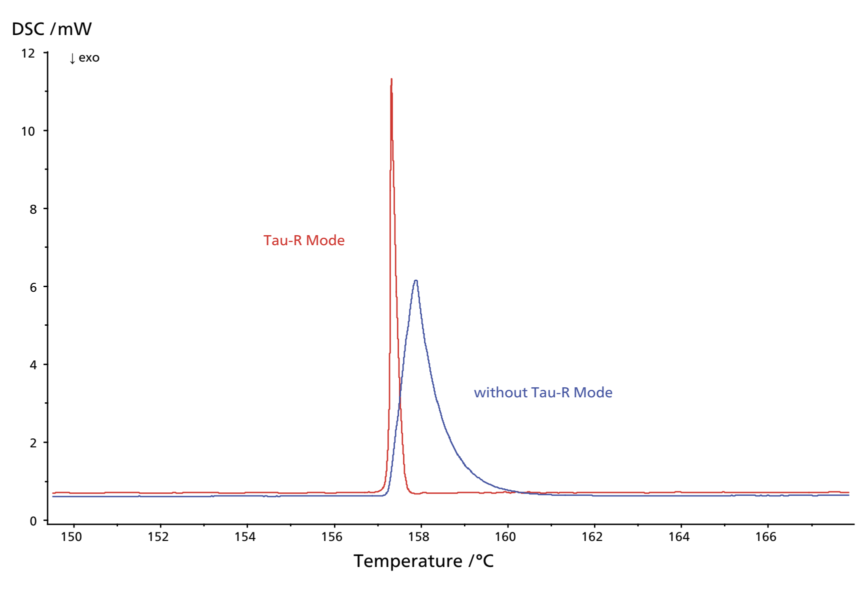
Key Features of the Analysis Software

- Comparison analysis of up to 64 curves/temperature segments from the same or different measurements
- Simultaneous analysis of multiple curves
- Loading of single files and simultaneous loading of multiple files (curve and parameter preview)
- Smoothing of the DSC curves with adjustable filter factors
- Calculation and presentation of the first and second derivative
- Value determination on a single curve or a curve family
- Curve subtraction of baselines and sample runs; subtraction of physically identical curves (DSC)
- Shifting of freely selectable segments to the origin (zero point) on the time axis to simplify comparison between multiple heating (or cooling) segments
- Re-import of measurements saved as ASCII-files
- Connection of segments by spline interpolation: Dynamic segments with the same heating direction and isothermal segments can be analyzed as interrelated and depicted temperature-scaled
- Import of standard tables of specific heat, freely configurable
- Storage of the analysis results and status for later restoration and continuation with analysis
- PIP graphical function (picture in picture), exchange of the full curve and excerpt graphics (FLIP) with further results evaluation
- Semi-automatic routines for the determination of characteristic temperatures (extrapolated onset, point of inflection, peak maximum, peak end)
- Fully automatic routine for complex peak evaluation (onset, point of inflection, peak, end point, peak height, peak width and peak area); calculation according to DIN or ISO method
- Determination of peak areas and partial peak areas using different baselines (linear, area-proportional, etc.)
- Determination of the specific heat c_p(T), optionally also in accordance with the ASTM method
- Integration of the c_p(T) curve for determination of the enthalpy of a reaction
- Evaluation routine for glass transition
- OIT evaluation
- Determination of the DSC integral using the curve of the partial peak areas (LF liquid fraction, solid fat index)
- Determination of the DSC conversion curve in % using the curve of the partial peak areas based on the enthalpy of a selectable reference substance
- Determination of the temperature-dependent crystallinity curve from partial peak areas and based on the enthalpy of a selectable reference substance

Proteus® Software Modules and Expert Software Solutions

The *Proteus*® modules and expert software solutions offer further advanced processing of the thermoanalytical data for more sophisticated analyses. The advanced software packages *Peak Separation*, *Purity Determination* and *Thermokinetics* are optionally available for both DSC versions. The *Proteus*® software extensions *Tau-R® Mode*, *BeFlat*® and *Specific Heat Determination* are optionally available for the DSC 404 ***F3*** *Pegasus*®. TM-DSC is exclusively available for the DSC 404 ***F1*** *Pegasus*®.



Correction of the thermal resistance and the time constant lead to defined peaks (*Tau-R® Mode*)

Tau-R® Mode

The DSC curve not only incorporates important information about the sample, but also residual information from the instrument. To correct for this established fact, the *Tau-R® Mode* has been developed. This method is based on two essential correction factors: the thermal resistance (R) and the time constant (τ). Determination and creation of a correction file must be carried out only once for identical measurement parameters. The true raw data can then be accessed by just one click of the mouse.

BeFlat®

Perfect thermal symmetry, which is often expected in a differential measurement set-up, is virtually impossible to realize in practice. The unique *BeFlat*® software corrects DSC baseline discrepancies attributable to thermal asymmetry by using a multi-dimensional polynomial dependent upon temperature and heating rate. *BeFlat*® removes the baseline discrepancies from the DSC signal and yields perfect horizontal DSC baselines with minimal deviations in the µW range. The true raw data signal can be re-accessed at any time with a simple click of the mouse.

Peak Separation

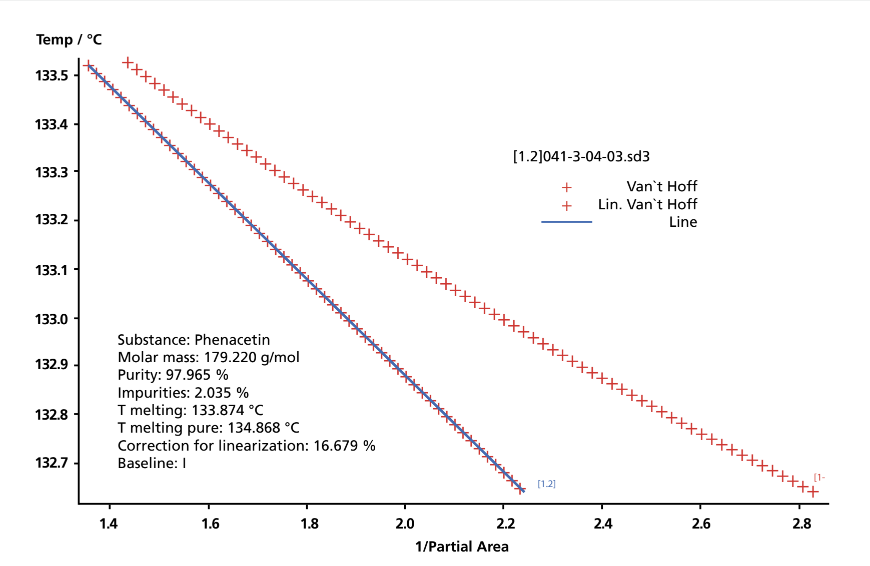
The *Peak Separation* software allows for the mathematical separation of overlapped peaks using profiles from the following peak types: Gaussian, Cauchy, pseudo-Voigt (linear combination of Gaussian and Cauchy), Frazer-Suzuki (asymmetric Gaussian), modified La-Place (double-sided rounded) and Pearson. The software fits the experimental data as an additive superposition of peaks.

Specific Heat Determination

The specific heat capacity (c_p) is an important temperature-dependent thermophysical property of a material. The DSC method allows for convenient and reliable determination of c_p. Knowledge of the specific heat capacity supports the improvement and development of many technical and technological processes, as well as that of a broad variety of materials from building materials to turbine plates. This software module is based on standardized methods described in standards such as ASTM E1269, ISO 11357, DIN 51007.

Purity Determination

The *Purity Determination* program is applied to determine the purity of a material by analyzing the DSC melting peak. The calculation is carried out using the Van 't Hoff equation and is based on the fact that the melting process shifts to lower temperatures and the DSC melting peaks broaden as the impurity content increases. A given application range could thus be extended, for example, beyond an impurity threshold of 5 mol% to an estimated upper boundary of 10 mol%. In addition, the thermal resistance is adjusted so as to calculate the nominal value for substances with a known degree of purity.



Purity results for phenacetin standard with a 2% aminobenzoic acid content

Temperature-Modulated DSC

Temperature-modulated DSC (TM-DSC) is a DSC technique whereby a sample is subjected to a superposition of a linear and a periodic temperature program. Temperature-modulated DSC enables the separation of overlapped DSC effects by calculating the reversing and non-reversing signals. Glass transitions can therefore be well separated from, for example, exothermal curing, decomposition, evaporation, relaxation or cold-crystallization processes in a single TM-DSC test.

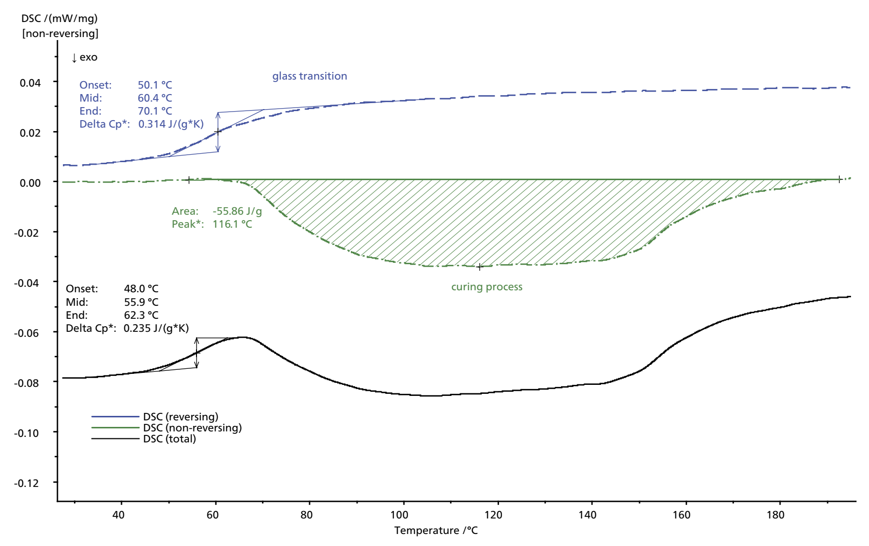
The DSC 404 ***F1*** *Pegasus*® is the only DSC which allows for TM-DSC tests up to 1650°C. Nearly all available furnaces in combination with different DSC sensors are suited for such tests. The NETZSCH TM-DSC technique is totally based on a software solution. The advantages can be summarized as follows:

- Offline evaluation after the measurement. This means there is no time delay of evaluated data, which is common for online evaluations
- FRC method which takes into account the dependence of the calibration coefficient on frequency, thermal resistance between the sample and crucible, and heat capacity of the sample.

This results in unique features which do not all require calibration with a c_p standard. These include:

- Unique baseline correction algorithm for average, amplitude and phase of the DSC curve
- Standard DSC calibrations (using melting points according to DIN 51007 and ASTM E967 or ASTM E968)
- Unique method of determination of the calibration coefficients for calculation of the reversing heat capacity (c_p standard required).

The NETZSCH TM-DSC solution improves separation of time-dependent events from the total heat flow. It supports the investigations of glass transition, vitrification, crystallization, evaporation, chemical reaction (e.g., curing), polymorphism and dehydration.



Curing of an unsaturated polyester resin and overlapping of an endothermal glass transition can be separated in a single TM-DSC test.

Thermokinetics

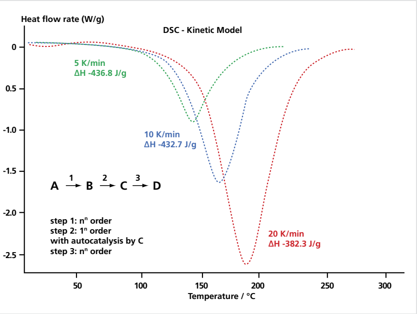
The *Thermokinetics* software module is used to create kinetic models of chemical processes based on a series of laboratory tests under different temperature conditions. It can also be used to predict the behavior of chemical systems under user-defined conditions for process optimization. *Thermokinetics* is capable of analyzing any process with a reaction rate which is a function of temperature and time.

Analysis with *Thermokinetics* makes it possible to determine the number of reaction steps and the following values for each step:

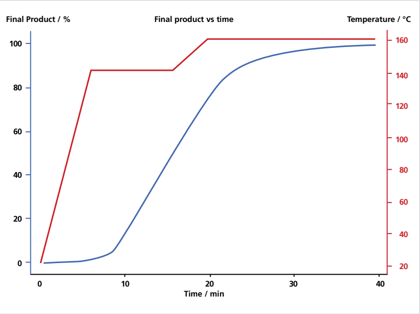
- Reaction type
- Activation energy
- Reaction order
- Other kinetic parameters

The program contains the following kinetic analysis and prediction methods:

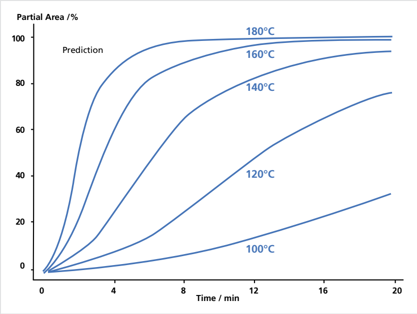
- Model-free kinetic analysis (Friedman, Ozawa-Flynn-Wall, ASTM E698, ASTM E1641)
- Model-fit using multivariate nonlinear regression (model definition, up to 6 multiple-step reactions, 18 different reaction types, F-test for fit quality and significance of an additional step)



Fitting of the DSC curves by using the 3-step reaction model



Prediction for the final product according to a temperature program

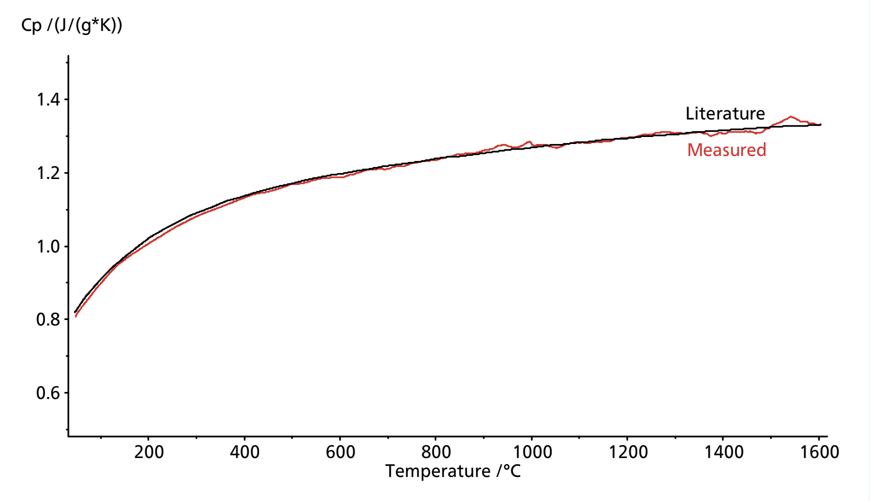


The fractional reaction is calculated for a set of temperatures and a certain time

Performance and Applications – Specific Heat Capacity, Phase Transitions

Accuracy of the Specific Heat Capacity of Alumina (Al_2O_3)

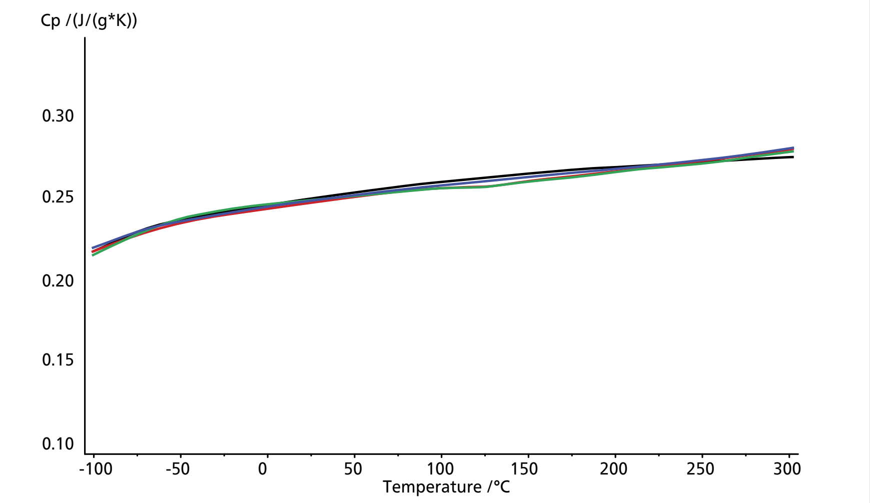
Presented in the figure is a specific heat capacity test result for a polycrystalline alumina sample between room temperature and 1600°C. Additionally shown are literature values for pure alumina. There are no significant differences between the literature values and the test results. The maximum deviations are in the range of 2%, which demonstrates the outstanding performance of the DSC 404 ***F1*** *Pegasus*®.



Highly accurate determination of the specific heat capacity across a wide temperature range. For these measurements, the rhodium furnace and the DSC-c_p sensor type S were used.

Determination of the Specific Heat Capacity of Molybdenum in the Low-Temperature Range

The specific heat of molybdenum was measured three times between -100°C and 300°C using the low-temperature furnace. The three results (colored lines) are identical to within 2%. Additionally shown are literature values for pure molybdenum (black line). The differences between the measurement results and the literature values are generally less than 2%. This proves the excellent specific heat performance of the system in the low-temperature range.



The specific heat capacity in the sub-ambient temperature range can be determined up to 300°C by means of the steel furnace (-150°C to 1000°C) and the DSC-c_p sensor type E.

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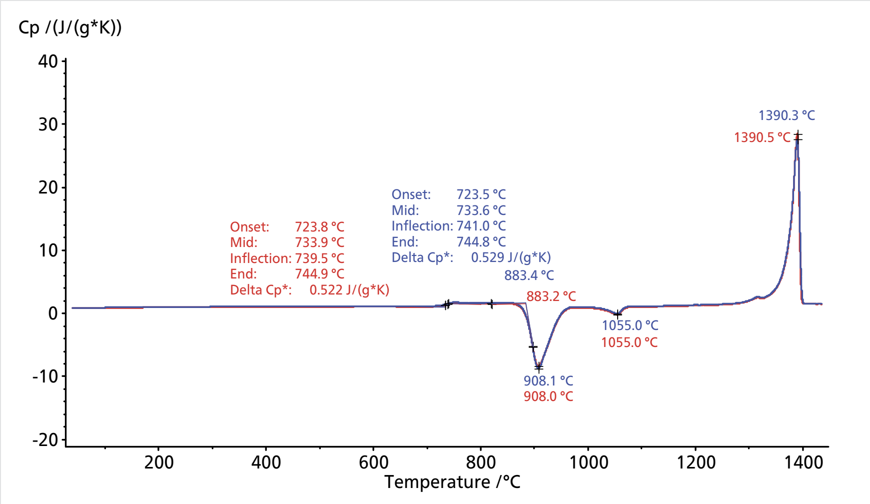
Reproducibility of the Apparent Specific Heat of a Diopside Glass Powder

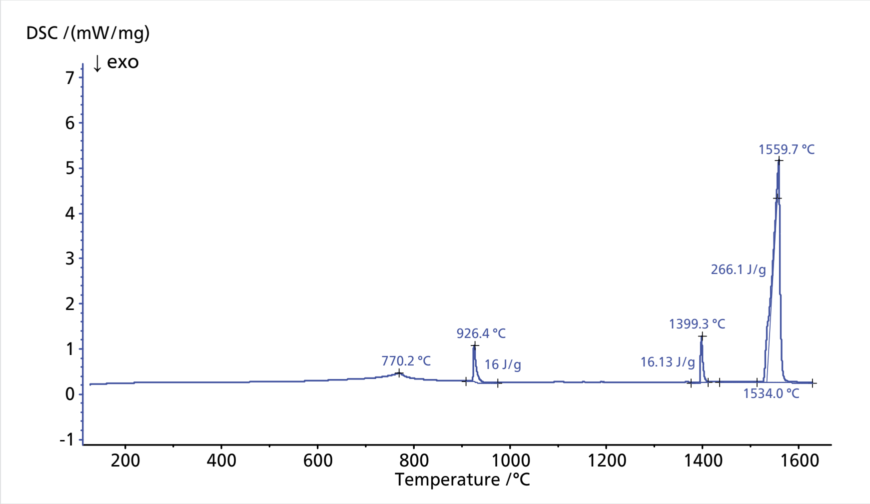
In this plot, the glass transition of the powder occurred at 734°C (midpoint). Crystallization was measured at 883°C (extrapolated onset). Melting took place at 1390°C (main peak temperature). The characteristic temperatures and the corresponding enthalpy changes are in good agreement between the two different samples. The specific heat itself doesn't show relevant differences, which indicates the excellent stability and reproducibility of the DSC system.



Excellent reproducibility and the detection of weak phase transitions (glass transition) can be achieved by simply combining the right furnace with the right sensor. These DSC runs were carried out with the Pt furnace and by using DSC-c_p sensor type S.

Phase Transitions of Iron

The specific heat flow rate of iron was here measured between room temperature and 1620°C. The peak at 770°C is due to a change in the magnetic properties of the material (the Curie transition). At peak temperatures of 926°C and 1399°C, two changes in the crystal structure occurred. Most likely due to impurities in the material, these temperatures are slightly shifted from the literature values [1] for pure iron. Melting occurred at 1534°C (extrapolated onset). The heat of fusion was 266.1 J/g. This is less than a 1.5% deviation from typical literature values for pure iron.



Sharp peaks, reliable peak areas and a stable baseline over a broad temperature range are the dominant attributes of results produced by the DSC 404 ***F3*** *Pegasus*® system equipped with the rhodium furnace.

[1] Das Techniker Handbuch, Grundlagen und Anwendungen der Maschinenbau-Technik, 15. Auflage, Herausgeber Alfred Böge, Vieweg Verlag, 1999

Performance and Applications – TM-DSC, Melting and Crystallization

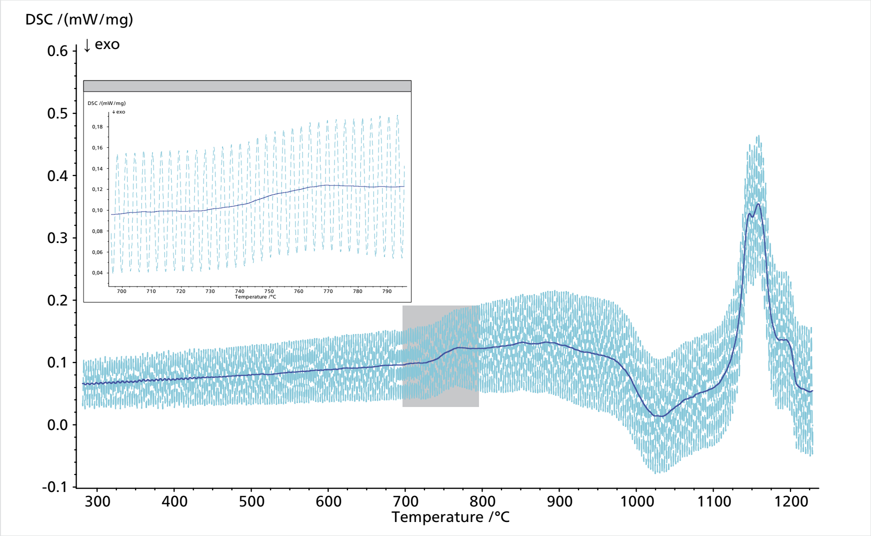
Temperature-Modulated DSC (TM-DSC) Measurement on a Ceramic Glass

For TM-DSC, the heating rates are varied by overlapping the underlying linear heating rates with a sinusoidal temperature modulation. At the same time, the sample is subjected to a non-linear temperature change which is relatively fast in comparison with the linear rate. These two conditions lead to the benefit of temperature-modulated DSC measurements: separation of reversing (thermodynamic) and non-reversing (kinetic) thermal effects.

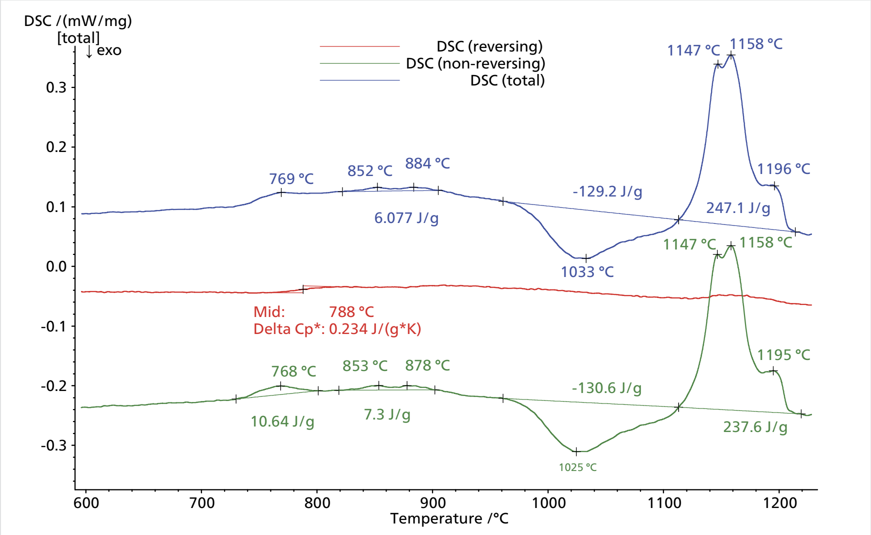
As shown with the example of a ceramic glass, the modulated signal (blue curve) is separated in a reversing (red curve) and non-reversing (green curve) signal. The reversible energetic effect can be seen from the reversing curve. Here, the glass transition was detected at 788°C. The relaxation peak was detected at 769°C in the total signal (blue curve) and especially in the non-reversing curve (green). Recrystallization and melting were observed as non-reversing signals at peak temperatures of 1030°C and 1158°C.

These results demonstrate that reversible and non-reversible effects – which may overlap in case of glass transitions and relaxation effects – can be separated by TM-DSC.

The non-reversing signal appears very similar to the total heat flow. It can be taken for kinetic studies and evaluation of the relaxation enthalpy and peak temperature.



The simplest evaluation of a typical TM-DSC measurement is the determination of the total heat-flow curve as an average value of the modulated heat-flow differential curve. Modulated signal (light blue curve) and average values (dark blue curve: total heat flow).



The TM-DSC measurement was carried out in a synthetic air atmosphere at a heating rate of 3 K/min and amplitude of 0.5 K/min for a period of 60 s. High-platinum crucibles with pierced lid were used. The sample mass amounted to 45 mg.

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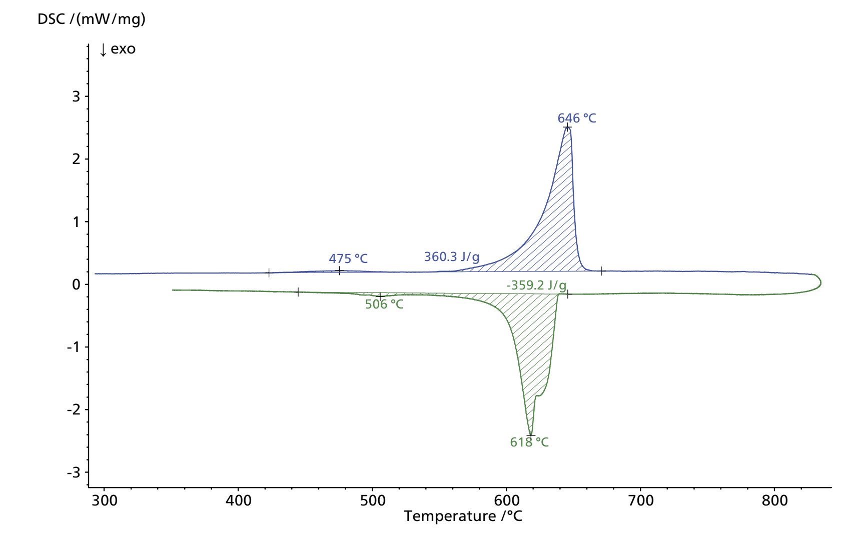
Melting and Crystallization of AlCuMg

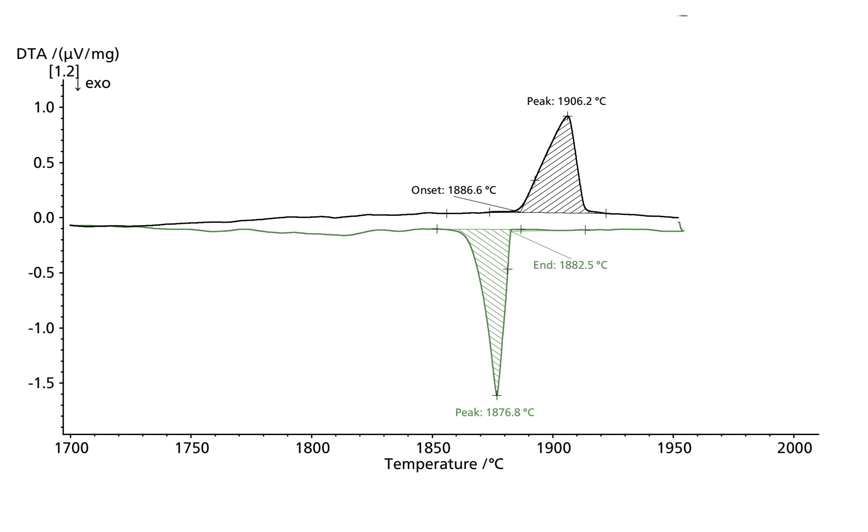
The DSC 404 ***F1/F3*** *Pegasus*® systems are able to perform controlled cooling runs. Here, the heating (blue curve) and cooling (green curve) runs on an AlCuMg alloy are presented. Melting occurs at 646°C (peak). Up to 800°C, no further thermal effects can be observed. Subsequent cooling leads to crystallization of the alloy. After a minor supercooling effect, crystallization reaches its maximum at 618°C. The melting and crystallization enthalpy are nearly identical. However, the shoulder in the crystallization peak already indicates crystallization of more than one structure.



Pt crucibles with a Al_2O_3 liner were used in order to prevent chemical reactions between the melt and the crucible material during a DSC measurement. Oxidation was avoided by using an argon atmosphere.

Melting Point Determination at Highest Temperatures

Determination of the melting and crystallization temperature up to 2000°C becomes possible by using the appropriate DTA sensor (type W/Re) and the graphite furnace. This example shows the heating (black curve) and cooling (green curve) runs on a vanadium sample (99.7 %). The DTA crucibles made of ZrO_2 are suitable in the highest temperature range. Melting occurs at an extrapolated onset temperature of 1887°C. During cooling, the sample recrystallizes with only a small supercooling effect at 1877°C (peak).



DTA measurement on vanadium in crucibles made of zirconium oxide (ZrO_2); heating rate 20 K/min, cooling 75 K/min helium atmosphere.

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