Thermal Analysis Excellence

Flash DSC 2+

STAR^{e} System

Innovative Technology

Versatile Modularity

Swiss Quality

Flash Differential Scanning Calorimetry

Gain New Insight into Materials

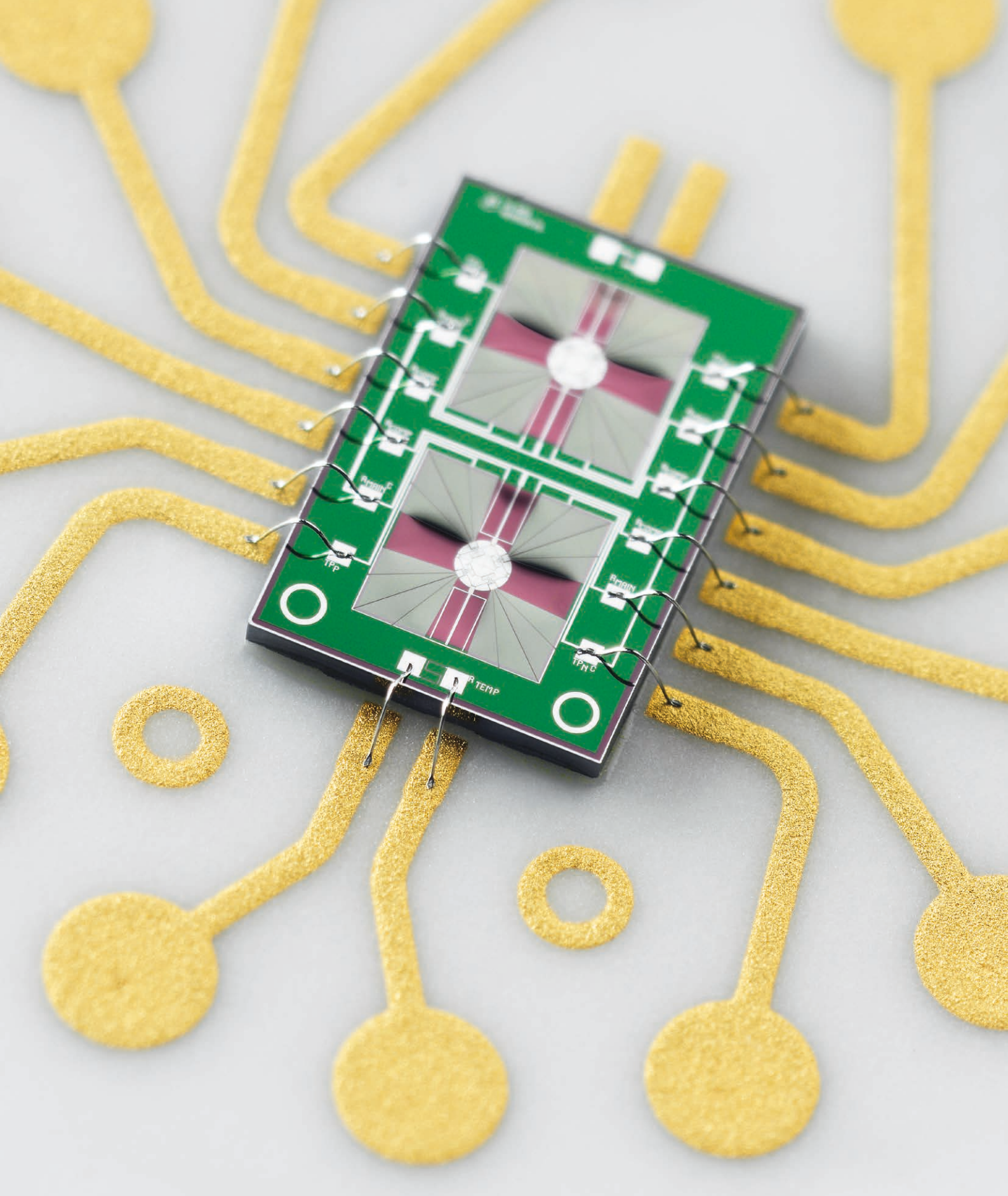
METTLER TOLEDO

Quantum Leap in Innovation
Opens up New Frontiers

Differential scanning calorimetry (DSC) is the most important method in thermal analysis. It measures the heat flow to or from a sample as a function of temperature or time and thereby allows physical transitions and chemical reactions to be quantitatively determined. The Flash DSC 2+ revolutionizes rapid-scanning DSC. The instrument can analyze reorganization processes that were previously impossible to measure.

Features and benefits of the METTLER TOLEDO Flash DSC 2+:

- **Ultra-high heating rates** – suppress reorganization processes
- **Ultra-high cooling rates** – allow the formation of materials with defined structural properties
- **High sensitivity** – permits measurements at low heating rates that overlap with conventional DSC
- **Wide temperature range** – perform measurements from –95 to 1000 °C
- **Gas-tight measuring head** – investigate samples under defined atmospheres
- **Fast response sensor** – enables the kinetics of extremely fast reactions or crystallization processes to be studied
- **Simplicity of use** – from easy sample preparation and sensor change to convenient result evaluation
- **Oxygen-free environment** – protect your sample against oxidation
- **User-friendly ergonomics** – for easy and quick sample preparation and sensor change



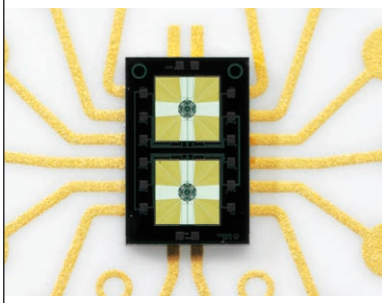
The MEMS technology-based chip sensors lie at the heart of the Flash DSC 2+.
(MEMS = micro-electro mechanical systems)

www.mt.com/ta-flashdsc

Unsurpassed Heating and Cooling Rates with Oxygen-Free Capability

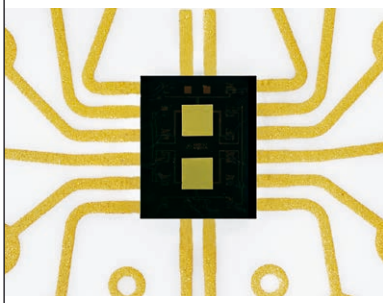
In contrast to conventional DSC, samples are placed directly onto the MultiSTAR™ chip sensor in the Flash DSC 2+, such that effects caused by the crucible material are eliminated. In addition, the patented dynamic power compensation control circuit allows measurements to be performed at high heating and cooling rates with minimum noise levels.

Standard
MultiSTAR UFS 1 sensor



The UFS 1 sensor is equipped with 16 thermocouples for high sensitivity and excellent temperature resolution. The MEMS chip sensor is mounted on a stable ceramic substrate with electrical connections.

High-temperature
MultiSTAR UFH 1 sensor



The newly developed UFH 1 sensor permits measurements in a wide temperature range from –95 to 1000 °C. The extremely short time constant, of about 0.2 ms, makes it possible to achieve much higher heating and cooling rates of 3'000'000 and 2'400'000 K/min, respectively.

Oxygen-free conditions

The wide operating temperature range allows transformations in various materials to be investigated. Some of them, for instance metals, react with oxygen (especially at high temperatures). Such reactions are avoided in the Flash DSC 2+, which permits samples to be measured under oxygen-free conditions.

The Flash DSC 2+ is operated using either of the two MultiSTAR sensors (UFS 1 or UFH 1) mounted on a stable ceramic substrate with electrical connections.

www.mt.com/ta-fdsc

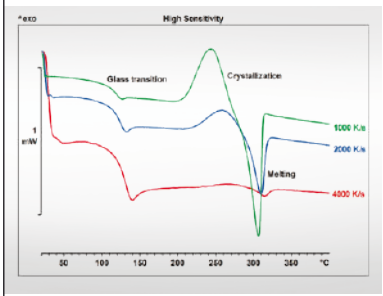
Swiss Quality

Flash DSC 2+ from METTLER TOLEDO for Maximum Accuracy

The Flash DSC 2+ allows you to prepare samples with defined structures such as occur during rapid cooling in injection molding processes. The application of different cooling rates influences the crystallization behavior and structure of the sample.

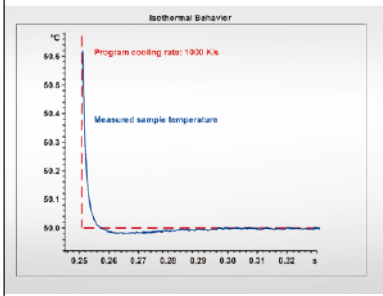
The use of high heating rates enables materials to be analyzed without interference from reorganization, as there is no time for such processes to occur. The Flash DSC 2+ is also the ideal instrument for studying crystallization kinetics.

Outstanding sensitivity



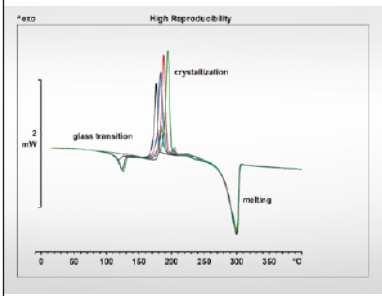
Two sets of 8 thermocouples, arranged symmetrically around the measurement area on the sample and reference sides of the UFS 1 sensor, deliver outstanding flexibility and permit temperatures to be measured with great accuracy, even at low heating and cooling rates.

Improved time constant



The time constant is fundamental for fast-scanning rates and isothermal measurements. The smaller the time constant, the easier it is to separate close-lying thermal effects. The time constant of the UFH 1 sensor is about 0.2 ms or about 5000 times less than that of a conventional DSC instrument.

High reproducibility



The MEMS-based technology of the chip sensor, alongside its excellent thermal contact with the sample, results in highly reproducible measurements, as noted in the melting area of the curve. Multiple measurement curves of differently prepared samples are thus easily compared.

The Flash DSC 2+ is the ideal complement to conventional DSC for characterizing modern materials and optimizing production processes by thermal analysis.

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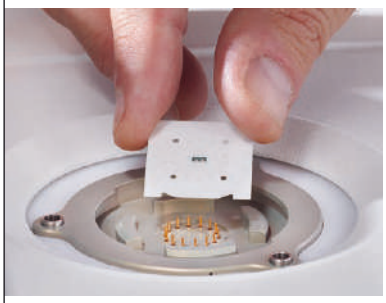
Simplicity of Use
Is What Customers Value

The preparation and insertion of a sample is performed while sitting comfortably in front of the instrument. The sample is first cut to size on a small glass microscope slide placed over the sensor. A suitable sample specimen is then transferred directly onto the sensor and positioned using a strand of hair.

Sample preparation

Sample preparation is carried out with the aid of a microscope. The microscope is also used to accurately position extremely small samples onto the sensor.

Reusable chip sensors



Central to the Flash DSC 2+ is the reusable chip sensors based on MEMS (micro-electro mechanical systems) technology. Sensors with the adhered sample can be changed in less than a minute and afterward safely stored in the chip sensor box supplied.

Touch-screen terminal

A large and easy-to-read color touchscreen displaying the status of your instrument is located at the front of the Flash DSC 2+. Individual sequences and queries may also be added directly via the touchscreen without the use of a PC.

Sensors are easier and faster to fix, and achieve results with higher reproducibility, thanks to the improved sensor clamping mechanism of the Flash DSC 2+.

Increased Efficiency
Thanks to Practical Accessories

The expanded temperature range of –95 to 1000 °C covers the most relevant materials. The Flash DSC 2+ can be cooled by IntraCoolers – electrical cooling devices with a closed-loop cooling system. The vaporized coolant is liquefied by means of compressors and heat exchangers.

Chip sensor boxes

Each reusable chip sensor is limited to just one sample. If measuring the same sample more than once, it is recommended to safely store individual sensors, with the adhering sample, in the chip sensor box.

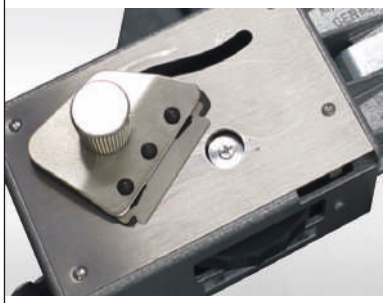
Blue box: UFS 1/ST sensors
Red box: UFH 1/HT sensors

Standard accessories

The following tools needed to prepare thin layers are supplied with the instrument as standard equipment:

- knife with spare blades
- lancet-shaped needle
- tweezers
- leather cloth
- grinding stone
- brush
- hair holder
- glass support and
- indium and aluminum for calibration

Microtome (optional accessory)



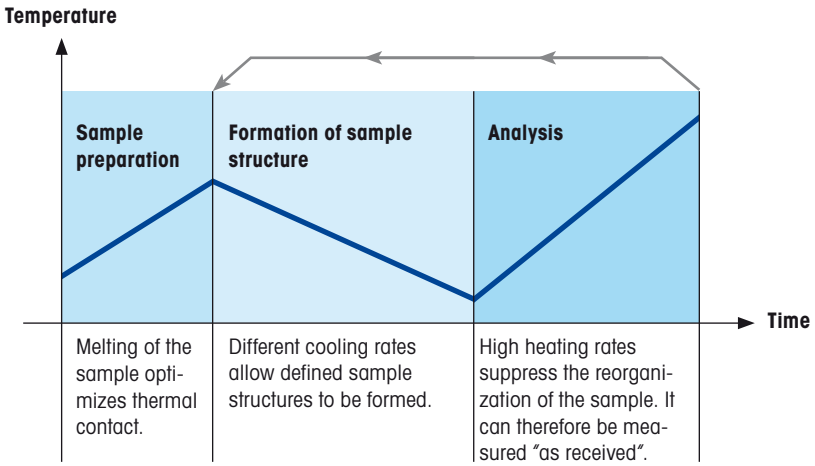
The microtome can be used to cut materials, such as a small polymer granule, into layers from 10 to 30 µm thickness. These layers are then prepared for analysis by cutting them into small sample specimens using the knife supplied.

The screw closure lid ensures oxygen-free conditions. It tightens the measuring cell and prevents any gas exchange with the surroundings.

Outstanding Performance
Leads to Novel Results

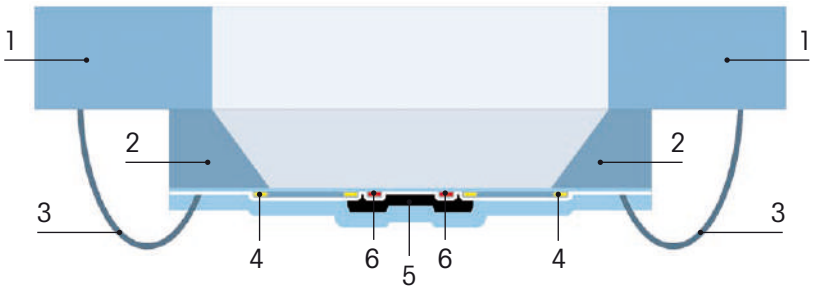
Measurement principle: High heating or cooling rates are only possible when the sample is sufficiently small and in good thermal contact with the sensor. The sample melts in the first heating run, which greatly improves the thermal contact. Defined sample structures can then be produced by varying the cooling rate over a wide temperature range.

In the second heating run, the sample has no time to reorganize because of the very high heating rates. The enormous range of cooling and heating rates allow many different sample structures to be measured in one experiment.

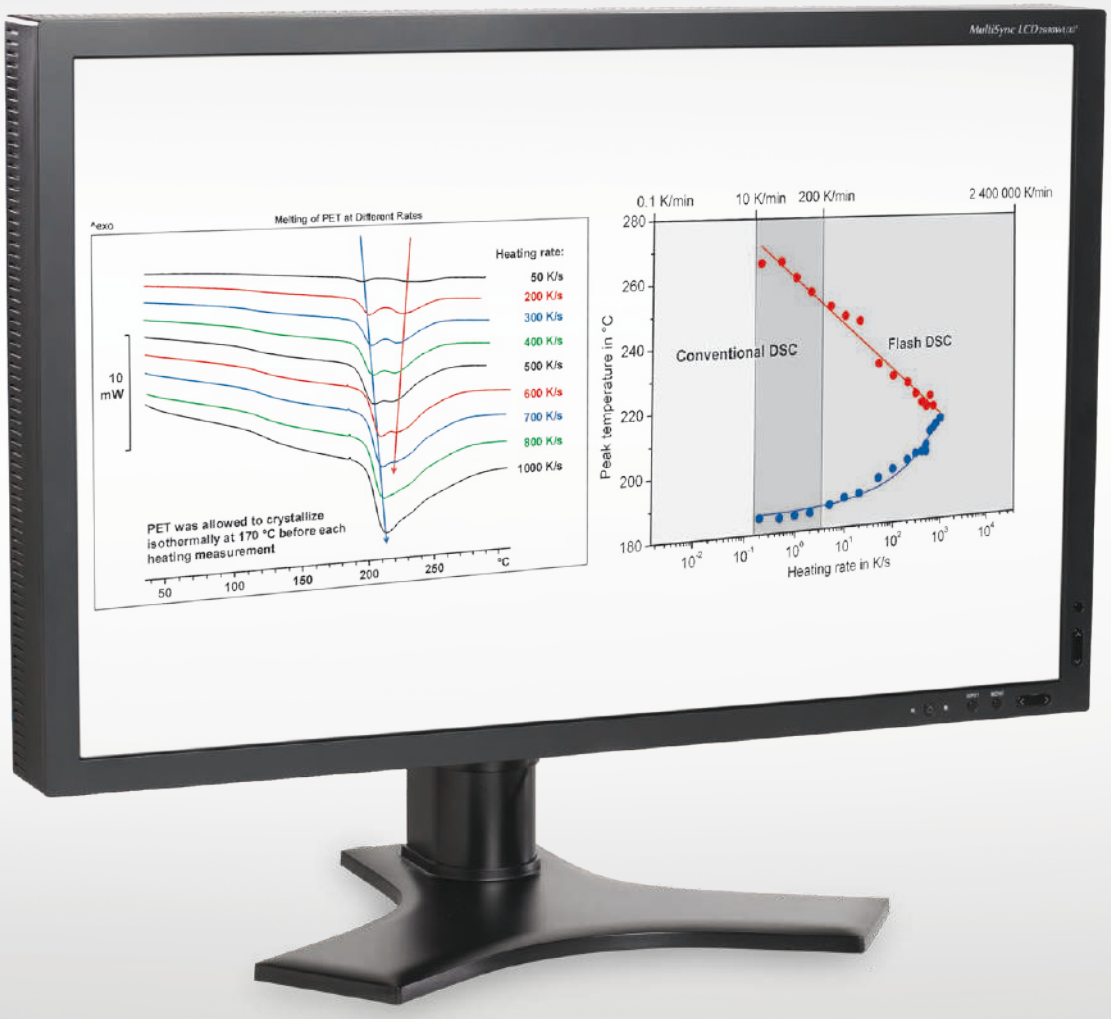


Chip sensor principle

The sample and reference sides of the UFS 1 sensor each have two thermal resistance heaters, which together generate the desired temperature program. The smaller heater is for compensation control (dynamic compensation control). The heat flow is measured using the two sets of 8 thermocouples arranged symmetrically around the measurement area on the sample and reference sides of the sensor.

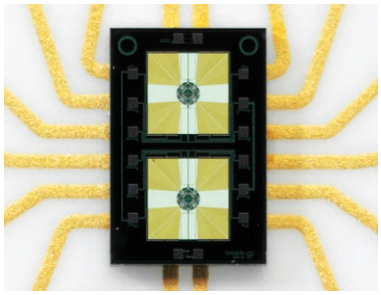


1. Ceramic plate
2. Silicon frame
3. Connecting wire
4. Resistance heater
5. Aluminum plate (sample area)
6. Thermocouple



Homogeneous temperature distribution

The sample measurement area of the chip sensor is made of silicon nitride and silicon dioxide coated with a thin layer of aluminum. This provides an extremely homogeneous temperature distribution across the sensor. The active measurement area is only approximately 2.1 µm thick. Therefore, the time constant is mainly determined by the sample.



Reorganization of polyethylene terephthalate (PET)

Many polymers exhibit reorganization effects in the DSC measurement curve when they are heated. The curve does not therefore show the melting of the crystallites originally present in the sample. This is demonstrated using a sample of PET that had been allowed to crystallize at 170 °C for 5 min before cooling to room temperature. The measurement curves at heating rates between 0.2 K/s and 1000 K/s show two peaks. The peak at lower temperatures shifts to higher temperatures with increasing heating rate (blue arrow). This peak occurs when the original crystallites melt.

The high-temperature peak shifts to lower temperatures (red arrow). This peak is due to the melting of crystallites produced through reorganization during the measurement. Only one peak is observed at 1000 K/s. Practically no reorganization occurs from this heating rate onward.

New Materials for the Future
Flash DSC Has the Answers

The Flash DSC 2+ is the ideal addition for characterizing modern materials and optimizing production processes by thermal analysis.

Polymers, polymorphic substances, many composites and blends have metastable structures that depend on the cooling conditions used in their production. On heating, reorganization processes such as the melting and recrystallization of unstable crystallites or the separation of phases may occur. The influence of reorganization on the heating curve can be analyzed by varying the heating rate.

Flash DSC can simulate technical processes in which rapid cooling occurs. This yields information about the effect of additives (e.g. nucleating agents) under near-process conditions. Isothermal measurements provide information on the kinetics of transitions and reactions that take place in a few seconds.

Fast measurements save time in the analysis and development of materials. The quality of products can be improved through knowledge of structure formation at actual process cooling rates. The data can be used for simulation calculations and to optimize production conditions.

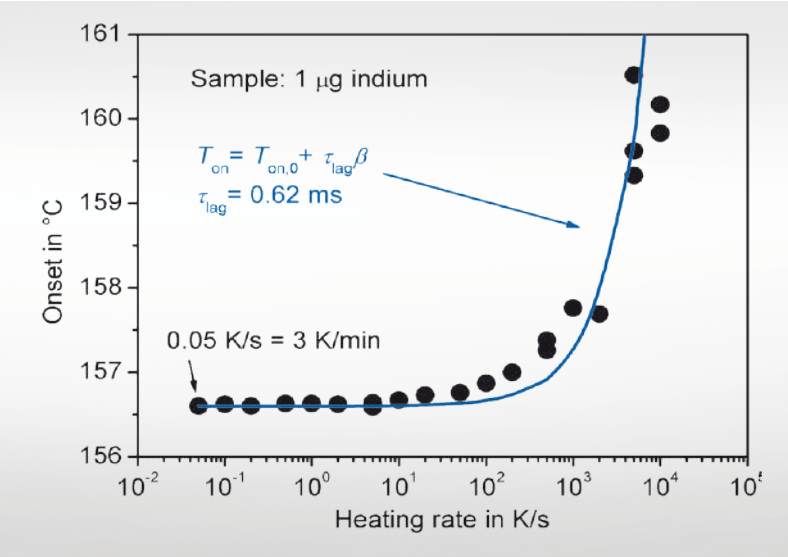
Application possibilities using the Flash DSC 2+

- Detailed analysis of processes involving the formation of structure in materials
- Direct measurement of rapid crystallization processes
- Determination of the reaction kinetics of fast reactions
- Investigation of the mechanism of action of additives under near-production conditions
- Comprehensive thermal analysis of materials in a very short time
- Analysis of very small sample amounts
- Determination of data for simulation calculations

The wide operating temperature range of the Flash DSC 2+ is ideal for the characterization of various metal alloys.

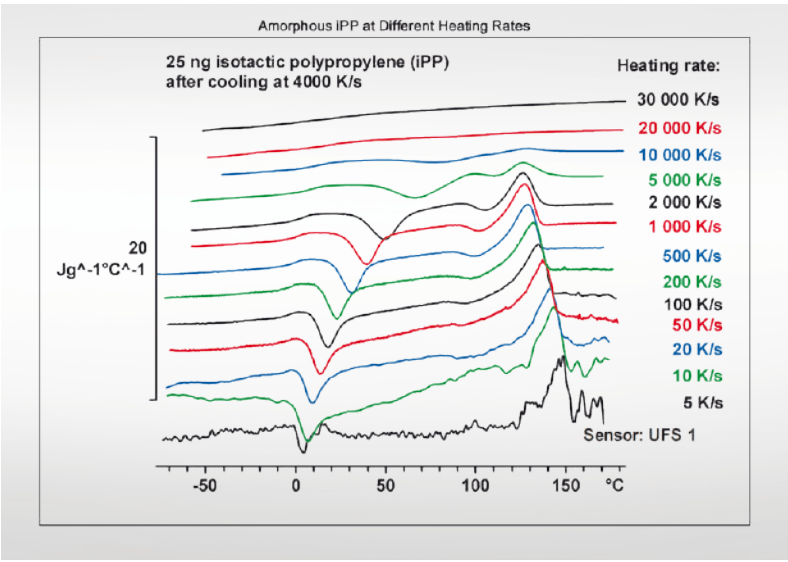
www.mt.com/ta-applications

Melting of indium at different heating rates



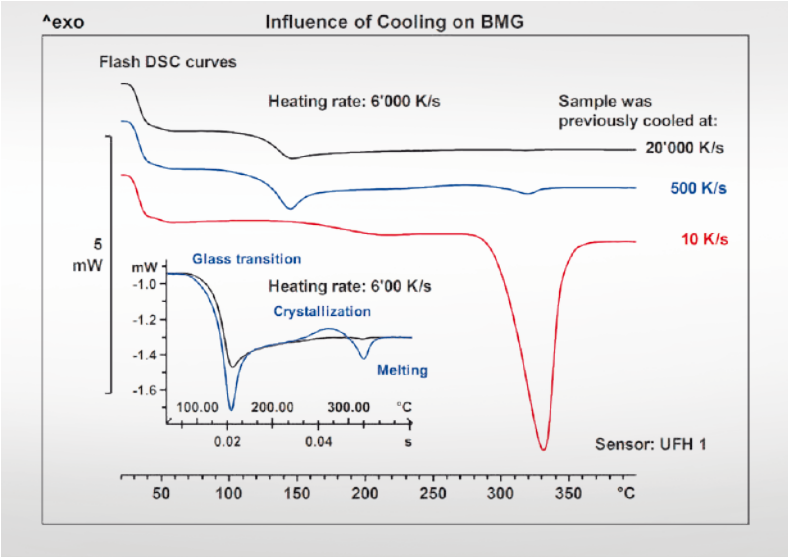
The melting of indium (1 µg) was measured at different heating rates between 0.05 K/s and 10'000 K/s using a UFS 1 sensor. As in conventional DSC, the conduction of heat between the sensor and the sample (thermal lag) influences the measured onset temperature, T_{on}. Without correction, T_{on} increases linearly with the heating rate. The same is true for the Flash DSC 2+. To accommodate the large heating rate range, the abscissa is displayed logarithmically in the diagram. For this reason, the linear function appears as a curve (blue).

Reorganization of amorphous isotactic polypropylene (iPP)



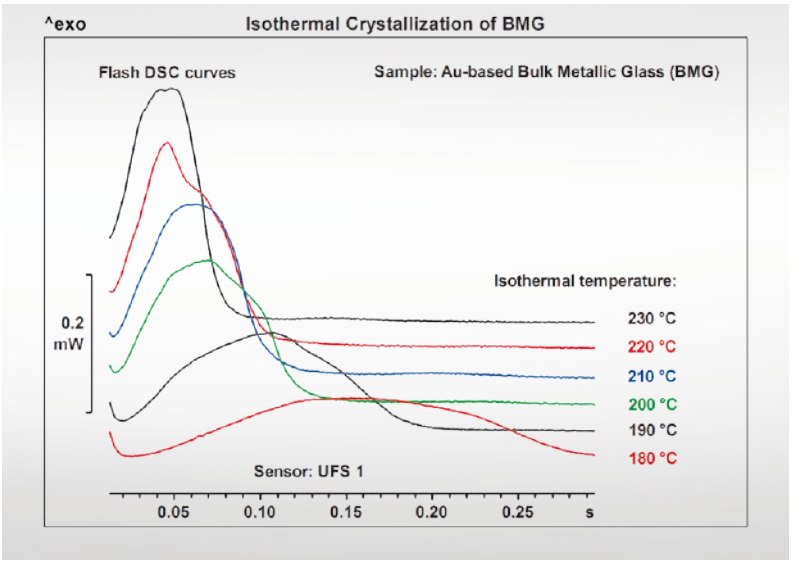
iPP is produced by cooling from the melt at 4000 K/s using a UFS 1 sensor. The material obtained was measured at heating rates between 5 K/s and 30'000 K/s. The glass transition occurs just below 0 °C followed by an exothermic peak due to cold crystallization. The crystallites melt above 100 °C. At higher heating rates, the cold crystallization peak is shifted to higher temperatures and the melting peak to lower temperatures. From 1000 K/s onward, the peak areas become significantly smaller until reaching 30'000 K/s, at which point reorganization in the sample no longer occurs.

Heating curves of an Au-based bulk metallic glass (BMG) alloy cooled at different rates



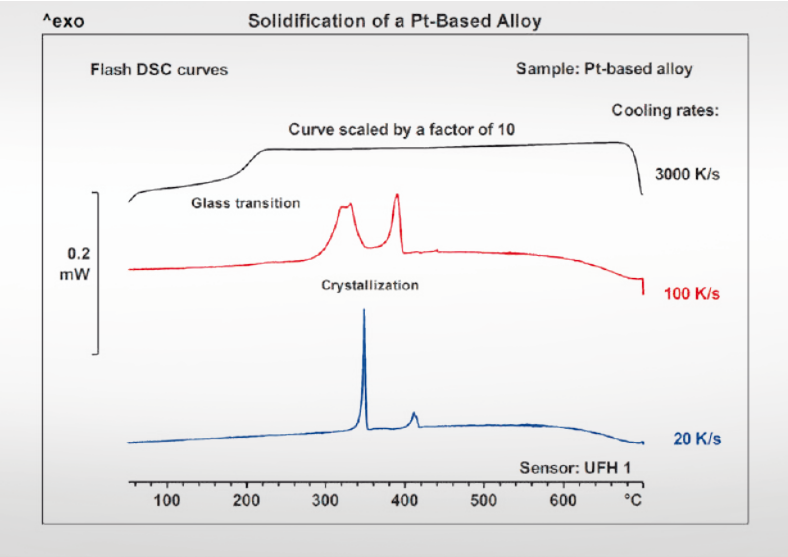
The figure shows heating curves of an Au-based BMG alloy measured at a heating rate of 6000 K/s. The sample was previously cooled in a Flash DSC 2+, equipped with a UFH 1 sensor, at 20'000, 500 and 10 K/s. After cooling at 10 K/s the sample is completely crystalline. The heating curve shows melting between 300 and 350 °C. The BMG alloy becomes amorphous after cooling at 500 and 20'000 K/s. The sample crystallizes slightly during heating, which explains the small melting peak. Both crystallization and melting events are magnified in the inset.

Isothermal crystallization of a BMG alloy



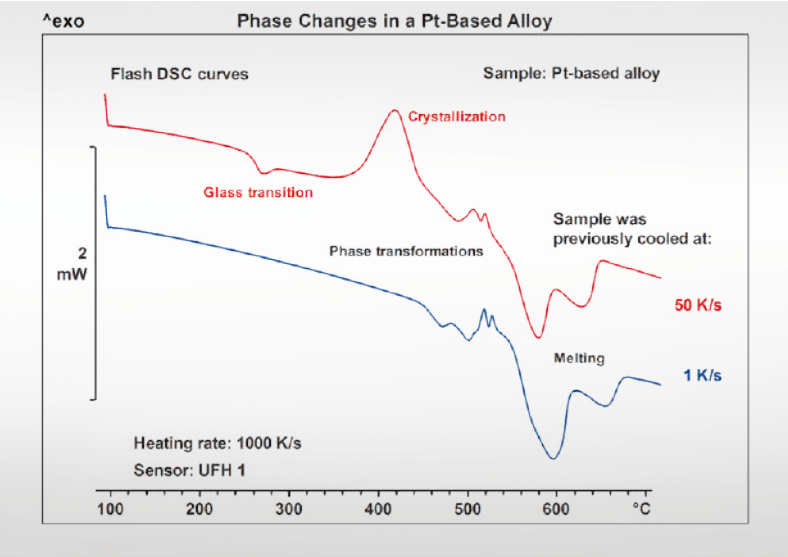
The short time constant of the UFS 1 sensor permits isothermal measurements in the time scale of milliseconds. The example shows isothermal crystallization curves after rapid heating at 30'000 K/s from the glassy state. Crystallization temperatures are given for each curve. The maxima of the crystallization peaks lie between approximately 40 and 160 ms.

Cooling curves of a Pt-based BMG



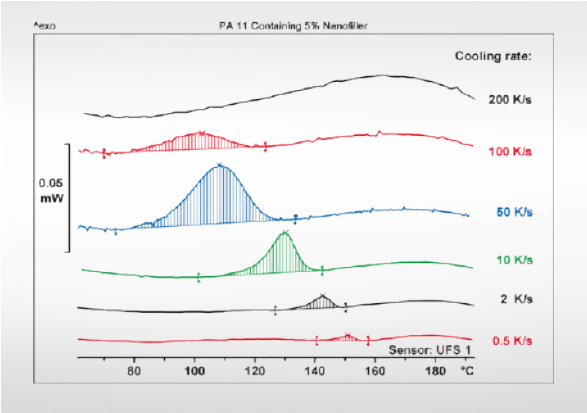
The figure shows the cooling curves of a Pt-based metal alloy cooled from 700 to 50 °C at different rates. The curve measured at 3000 K/s was divided by 10 to permit the representation of all heat flow curves in the same diagram. At relatively low cooling rates (20 and 100 K/s), two crystallization peaks are observed. The sample forms an amorphous glass when cooled at 3000 K/s.

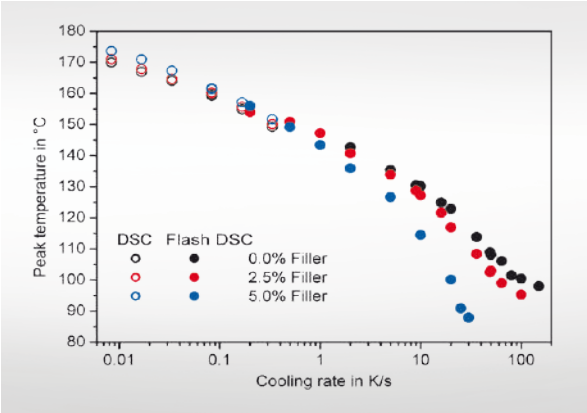
Crystallization, solid-solid transitions and melting of a Pt-based alloy

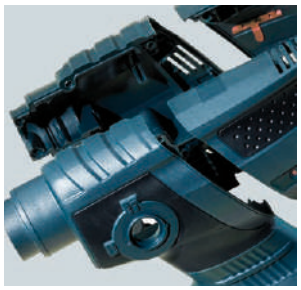


A metal alloy was previously cooled from the melt at 1 and 50 K/s. The slowly cooled alloy (1 K/s) exhibits solid-solid transitions below 580 °C when heated at 1000 K/s. At higher temperatures the sample melts. The faster cooled sample (50 K/s) is initially amorphous. The red curve shows the glass transition at about 250 °C, followed by crystallization.

Nanofillers in polyamide 11 (PA 11)

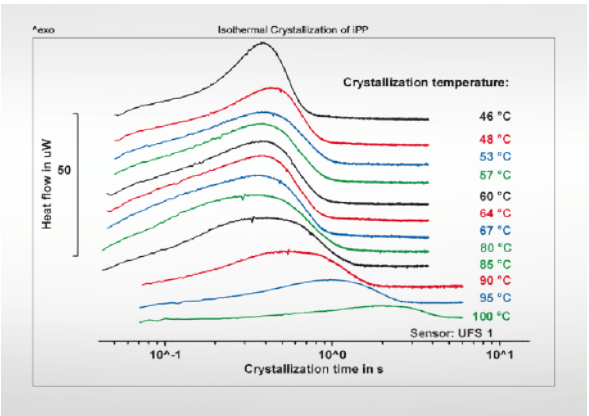


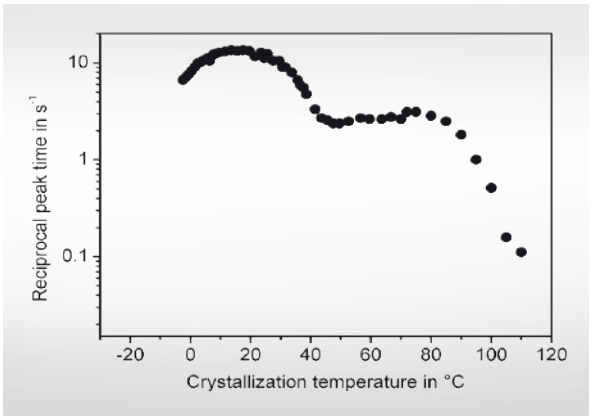


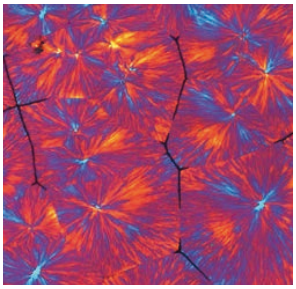


The properties of PA 11 can be optimized through the addition of nanoparticle fillers and the use of suitable cooling rates that mimic real processing conditions (e.g. for the injection molding of gearwheels). At such cooling rates, the fillers influence the size of the crystallites and hence the mechanical properties. Three PA 11 samples with nanofiller contents of 0%, 2.5% and 5% were measured at different cooling rates both in a Flash DSC and conventional DSC instrument. The enthalpy of crystallization at low cooling rates is constant up to 50 K/s, but becomes smaller at higher cooling rates. At 200 K/s, the sample no longer crystallizes. The influence of the cooling rate on the filler becomes clear when the peak temperatures are displayed as a function of the cooling rate. At cooling rates below 0.3 K/s (20 K/min), the unfilled PA 11 crystallizes first. In contrast, at higher cooling rates, characteristic of process-like conditions, crystallization is accelerated by the presence of nanoparticles.

Isothermal crystallization of iPP





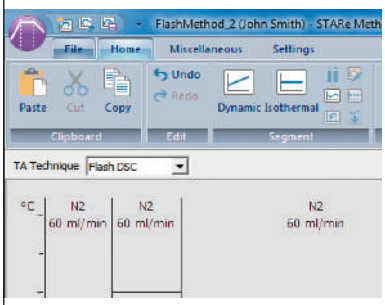


To measure the isothermal crystallization behavior of isotactic polypropylene (iPP), the melt was first cooled to different crystallization temperatures between 110 °C and –20 °C at 2000 K/s. No formation of structure occurs under such conditions. Afterwards, the crystallization process was measured isothermally. The exothermic crystallization peaks reach a maximum between 0.05 and 10 s. The reciprocal peak time is a measure of the crystallization rate and is displayed as a function of the crystallization temperature. The resulting curve exhibits a maximum at about 20 °C. At these low temperatures, crystallization takes place very rapidly via homogeneous nucleation. The measurement curves also demonstrate the variation of crystallization kinetics with temperature.

Simple, Intuitive Operation
Straightforward, Efficient and Secure

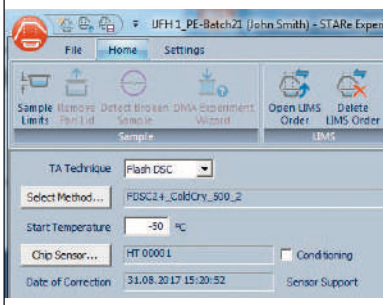
STARe software has been expanded to include new features that help you prepare your Flash DSC 2+ instrument for specific experiments, develop methods for advanced analyses and perform flexible result evaluations. Complex measurement programs are set up within minutes and the vast range of available tools permit curves to be evaluated both accurately and efficiently.

Graphical method development



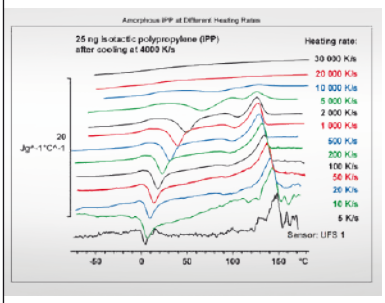
Any type of temperature profile, with up to 200 temperature segments, can be programmed in the Method Window of STARe software. A large number of complex operations relevant to Flash DSC – such as loops or conditional termination – contribute to well-designed experiments that help produce accurate results.

Experiment Window



The Experiment Window allows you to select a method and enter the relevant data for the specified experiment. Such data, in a typical Flash DSC experiment, includes the conditioning and correction procedure for the new sensor.

Benchmark for flexible evaluations



The Evaluation Window included in the base software offers the possibility of advanced evaluation tools, such as the Mathematics option, and a superior layout program aimed specifically at Flash DSC users. Such unrivalled flexibility puts your creativity at the forefront of the evaluation process.

A complete thermal analysis system consists of the basic six complementary measuring techniques, each of which bring fast and accurate results. Additional knowledge can be obtained by mewans of several hyphenated techniques.

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Flash DSC 2+ Specifications

Temperature data		
Temperature range	Air cooling	RT to 1000 °C (UFH 1) / RT to 500 °C (UFS 1)
	IntraCooler (1-stage)	–35 to 1000 °C (UFH 1) / –35 to 450 °C (UFS 1)
	IntraCooler (2-stage)	–95 to 1000 °C (UFH 1) / –95 to 420 °C (UFS 1)
Cooling rates (typical)		UFH 1: 6 to 2'400'000 K/min (or 0.1 to 40'000 K/s)
		UFS 1: 6 to 240'000 K/min (or 0.1 to 4'000 K/s)
Heating rates (typical)		UFH 1: 6 to 3'000'000 K/min (or 0.1 to 50'000 K/s)
		UFS 1: 6 to 2'400'000 K/min (or 0.1 to 40'000 K/s)
Sensor data		
Sensor membrane material		Silicon/silicon nitride (UFH 1) / silicon nitride (UFS 1)
Number of thermocouples		4 (UFH 1), 16 (UFS 1)
Signal time constant		Approx. 0.2 ms (UFH 1); approx. 1 ms (UFS 1)
Applied sample mass range UFS 1		Approx. 5 to 400 ng (org. materials, polymers); 100 to 10'000 ng (metals)
Applied sample mass range UFH 1		Approx. 5 to 100 ng (org. material, polymers); 50 to 5'000 ng (metals)
Flash DSC sensor		
Sensor types		UFH 1 (high-temperature) or UFS 1 (standard)
P_{max} heat flow signal		±20 mW
Noise heat flow signal		rms < 0.5 µW (typical)
Isothermal drift heat flow signal		< 5 µW/h (typical)
Gas conditions for measurement		
Oxygen-reduced atmosphere		< 50 ppm O_2
Terminal		
Touch control		Color TFT; WVGA 7" 800 x 480 pixel
Signal detection		
Sampling rate		Max. 10 kHz (10'000 points per second)
Resolution of temperature signal		7.5 mK (UFH 1), 2.5 mK (UFS 1)
Noise temperature signal		rms < 0.01 K (typical)
Communication		
With personal computer (PC)		Ethernet
Dimensions		
Instrument dimensions (width * depth * height)		45 * 60 * 50 cm

Approvals

IEC/EN 61010-1, IEC/EN61010-2-010 and IEC/EN61010-2-081
CAN/CSA C22.2 No. 61010-1, No. 61010-2-010 and No. 61010-2-081
UL Std. No. 61010-1
IEC/EN61326-1 (class B)
IEC/EN61326-1 (industrial requirements)
FCC, Part 15, class A
AS/NZS CISPR 11, AS/NZS 61000.4.3

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