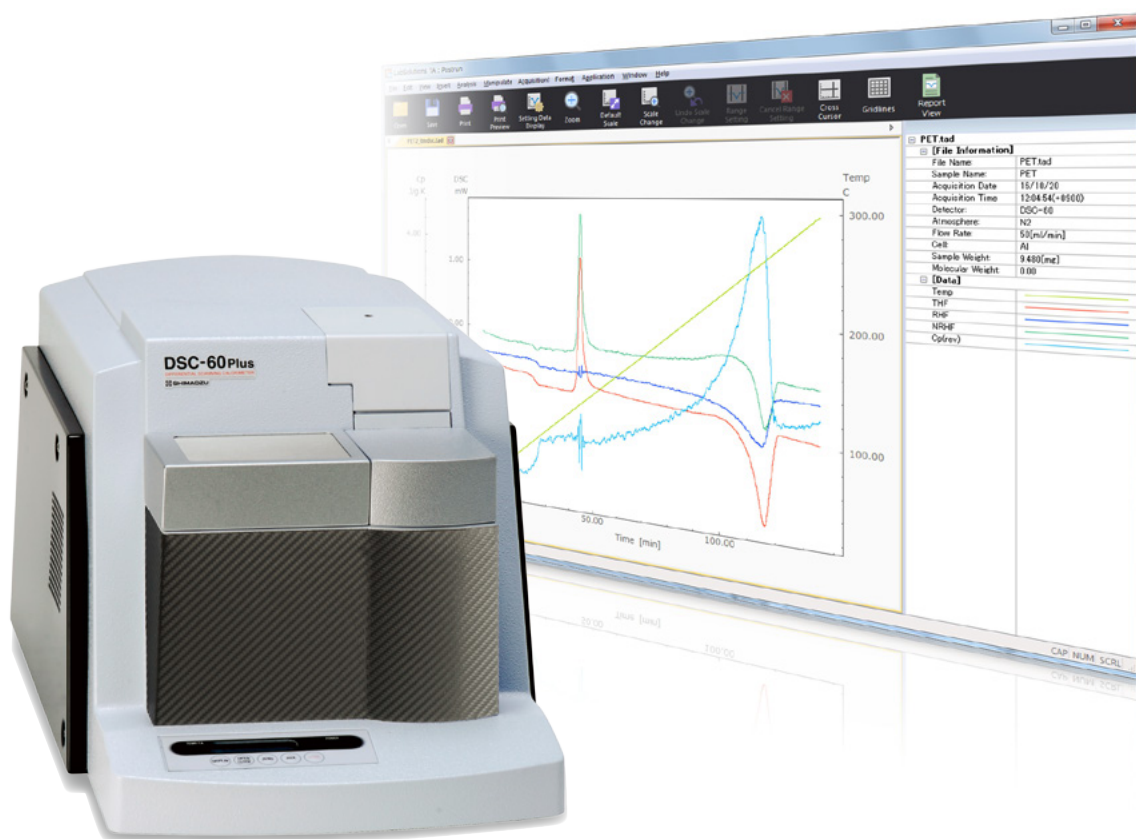


TM-DSC System for the DSC-60 Plus Series

Temperature Modulated DSC (TM-DSC) System



Features of the TM-DSC System

► Capable of Separating Complex Data Involving Overlapping Transitions and Reactions

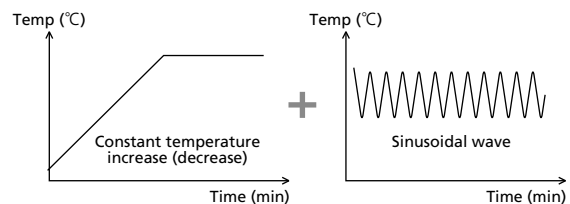
If glass transitions, enthalpy relaxations, and other reactions overlap, separation is impossible with standard DSC measurements. Using TM-DSC, however, enables separation of these phenomena.

► Easier Specific Heat Measurements

Specific heat measurements are easier than with standard DSC measurements

■ What Is TM-DSC?

By overlaying temperature-modulated results onto results obtained by increasing the temperature at a constant rate, TM-DSC can easily measure specific heat, or can measure multiple phenomena that could not be separated using standard DSC systems. This program can control the temperature by overlaying a sinusoidal modulation over a constant temperature increase (or decrease).



Three types of heat flow data can be obtained by post-measurement data analysis.

(1) DSC Signal (Total)

Indicates the total heat flow.

This is the same as the heat flow obtained from standard DSC systems. It is obtained by deleting the modulated component from the data obtained by TM-DSC.

(2) DSC Signal (Reversing)

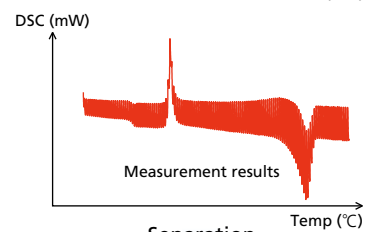
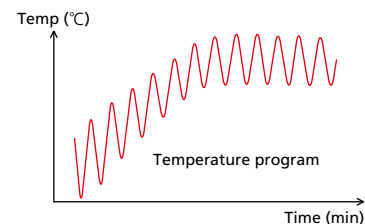
Indicates the reversing heat flow.

This component of the heat flow tracks the temperature modulation, and is equivalent to measured heat. It is obtained from the modulated component of data obtained by TM-DSC.

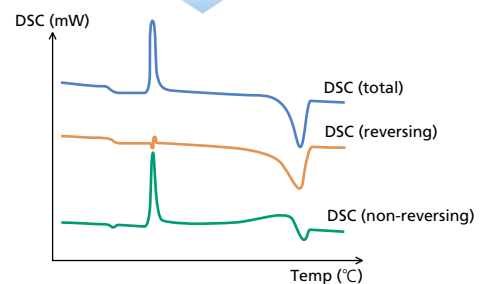
(3) DSC Signal (Non-Reversing)

Indicates the non-reversing heat flow.

This is the component of the heat flow that does not track the temperature modulation. It mainly appears for thermal processes that progress spontaneously, such as crystallization, curing, and enthalpy relaxation. It is obtained by subtracting the reversing signal from the total signal.



Separation



■ TM-DSC Functions

1 Separation of Glass Transitions

This program can separate reversing phenomena and non-reversing phenomena with respect to the temperature modulation.

Standard DSC: The reversing component and non-reversing component overlap.

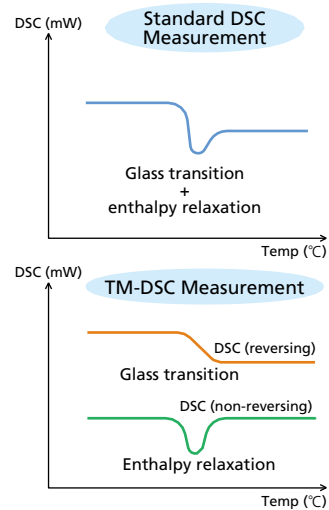
TM-DSC : The reversing component appears as the DSC signal (reversing), and the non-reversing component appears as the DSC signal (non-reversing), so they can be separated.

Example: Observation of an Overlapping Glass Transition and Relaxation Phenomenon

When measurements are performed with temperature modulation, the change in heat capacity due to glass transition appears in the DSC signal (reversing), whereas the endothermic peak due to enthalpy relaxation appears in the DSC signal (non-reversing).

This is also useful for the following measurements.

- Observation of recrystallization peaks during fusion
- Separation of the crystallization peaks for components in a polymer blend from the glass transitions of other components
- Observation of glass transitions in dehydration peaks
- Observation of glass transitions and the measured heat component of the heat flow in the curing process



2 Specific Heat and Heat Capacity Measurements

Measurements are easier to perform than with standard DSC.

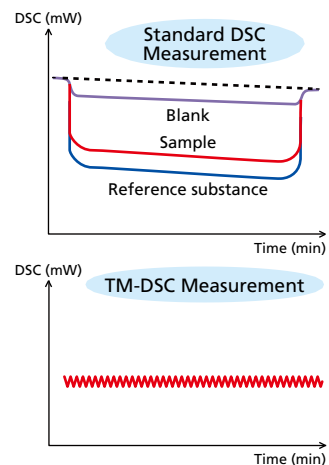
With TM-DSC, the specific heat is calculated in one cycle, so there is no measurement error due to baseline drift, and the results are accurate.

Standard DSC: Three measurements (the blank cell, the reference substance, and the sample) are performed.

TM-DSC : A single measurement of the sample is performed.

This System can display the reversing component of specific heat in terms of complex numbers and their phase.

It can also display the non-reversing component of specific heat.



3 Specific Heat Measurements Under Quasi-Isothermal Conditions

With TM-DSC measurements, the specific heat can be obtained as a function of time.

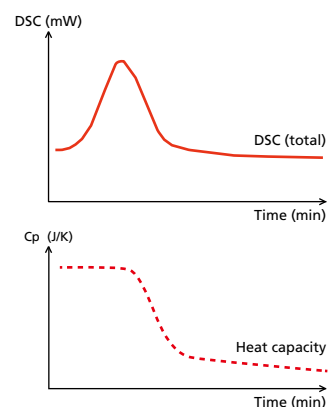
Consequently, this can be used to isolate changes over time, such as during curing, crystallization, or glass aging processes, something that was impossible with standard DSC measurements.

Standard DSC: Specific heat measurement can only be performed during processes with a constant temperature increase.

TM-DSC : Specific heat can be measured from the modulated component while keeping the temperature constant.

Example: Measuring the Curing Reaction of Epoxy Resin

When quasi-isothermal measurements are performed with TM-DSC, it is possible to assess the exothermic reaction due to curing, and at the same time, the change in the specific heat before and after the reaction.



Total Support from Measurement to Analysis

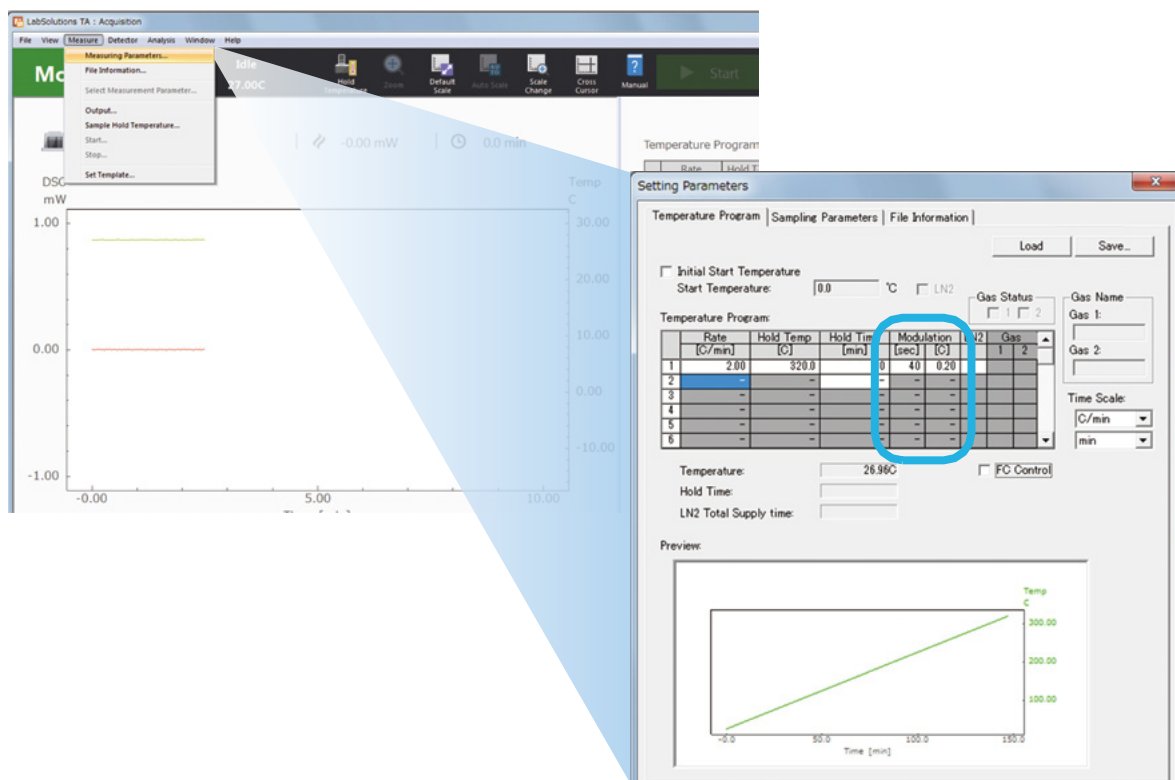
Step 1 Measure the Substance for Calibration.

Calibration data is obtained in advance in order to perform TM-DSC analysis.

Alumina powder is used as the sample for calibration measurement, which is measured under the same conditions as the sample.

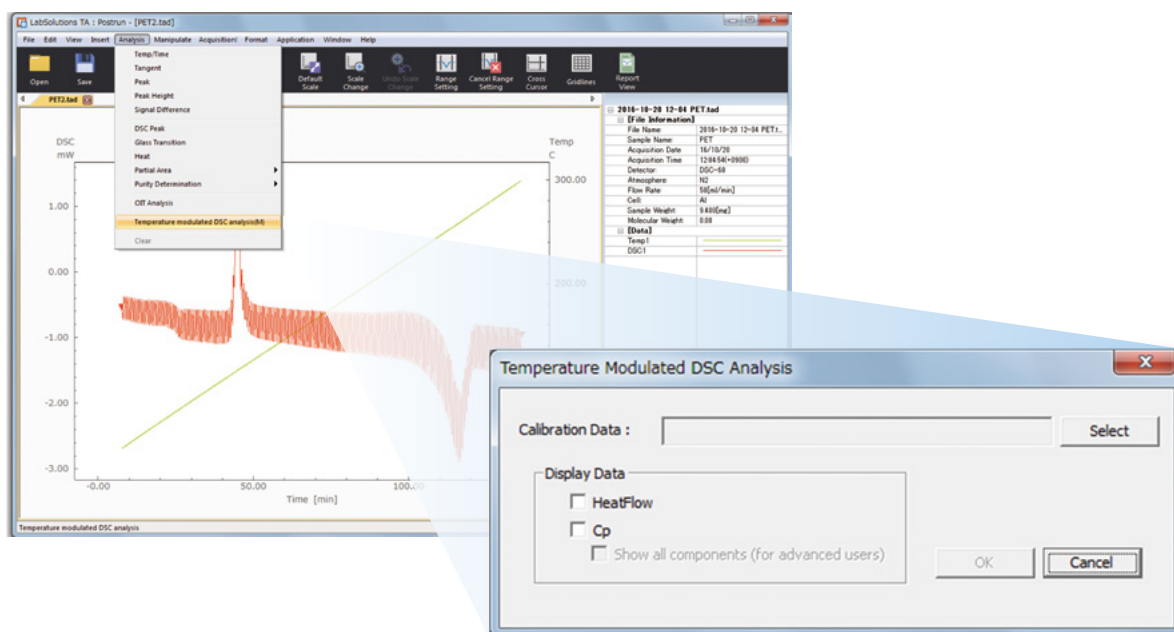
Step 2 Measure the Sample.

The sample is measured



Step 3 Analysis

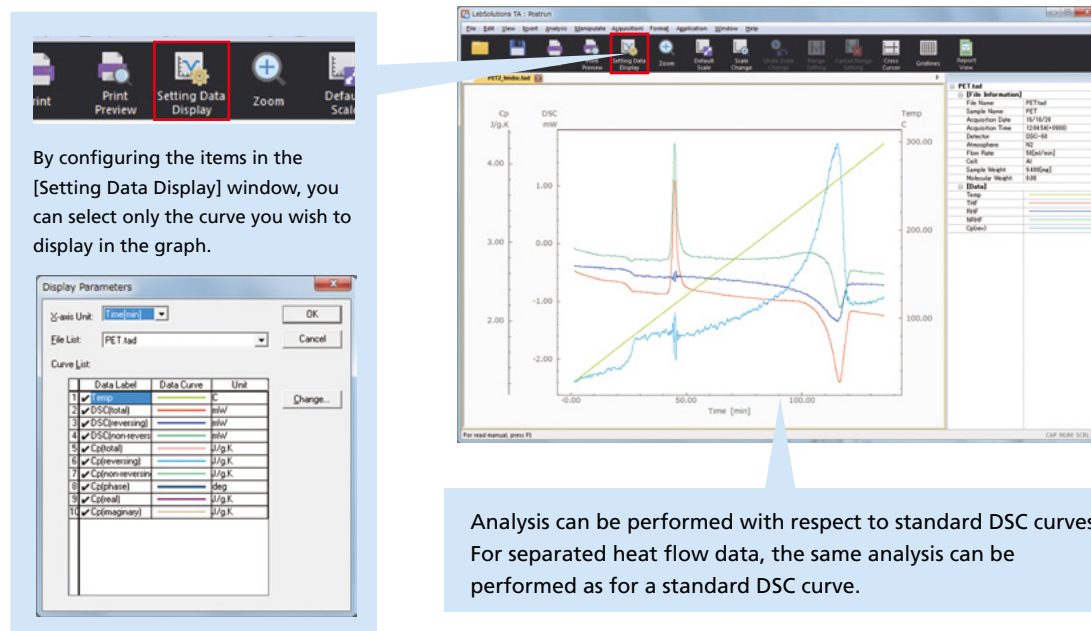
Analysis can be easily performed in the [Temperature Modulated DSC Analysis] window.



In the [Calibration Data] field, select the alumina measurement file.

In the [Display Data] area, select the type of data to display.

The type of graph to display can be changed later.



Analysis can be performed with respect to standard DSC curves. For separated heat flow data, the same analysis can be performed as for a standard DSC curve.

Applications

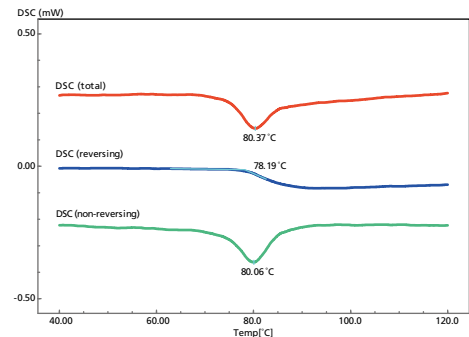
Measurements that were difficult with standard DSC can be performed. These include observation of glass transition points in polymer materials, observation of recrystallization occurring just before fusion, and the measurement of specific heat in quasi-isothermal measurements.

Separation of the Heat Flow for Impact-Resistant Polystyrene

With standard DSC measurements, the data for the glass transition and enthalpy relaxation near 80 °C overlap.

When temperature-modulated measurements are performed, the glass transition appears in the DSC (reversing) data, and the enthalpy relaxation appears in the DSC (non-reversing) data, so both can be separated.

In standard DSC measurements, the signals for phenomena that occur at basically the same temperature overlap, making analysis difficult. In contrast, when TM-DSC is performed, the signals can be separated into the reversing component and the non-reversing component.

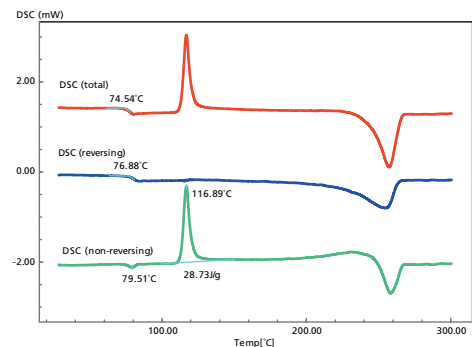


Separation of the Heat Flow for Polyethylene Terephthalate

With standard DSC measurements, the data for the glass transition and enthalpy relaxation near 75 °C overlap.

When temperature-modulated measurements are performed, the glass transition appears in the DSC (reversing) data, and the enthalpy relaxation appears in the DSC (non-reversing) data, so both can be separated.

In addition, the exothermic peak due to crystallization near 115 °C only appears in the DSC (non-reversing) data. At 200 °C and after, heat generation is seen in the DSC (non-reversing) data, so it is evident that recrystallization is occurring simultaneously during fusion.



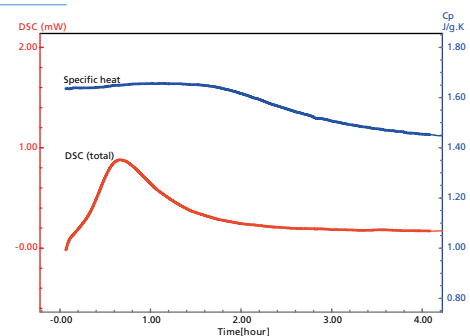
Quasi-Isothermal Measurements of an Epoxy Resin Adhesive

Quasi-isothermal control refers to thermal control with a modulation component added to the isothermal control that maintains a constant temperature.

With TM-DSC measurements, the specific heat can be obtained as a function of time, so the change over time in the specific heat resulting from chemical reactions progressing under constant temperature conditions can be seen. This could not be performed using standard DSC specific heat measurements.

When the adhesive was maintained under quasi-isothermal conditions at 100 °C, heat generation due to the curing reaction was seen immediately after measurement.

In contrast, from looking at the specific heat data, it could be confirmed that after the exothermic reaction, the change in the specific heat transitioned to a gentle decline. From this, it is evident that the epoxy undergoes glass transition as it cures.



System configuration



1	DSC-60 Plus (TAC/L)
2	LabSolutions™ TA
3	printer
4	Intracooler IP-100
5	Electric Auto-cooling Attachment TAC-60i
6	Temperature Modulated DSC(TM-DSC) Program
7	Flow controller FC-60A
8	Sample Sealing Crimp Press SSCP-1
9	Crimp attachment

Specifications

Applicable Models	DSC-60 Plus, DSC-60A Plus
Output Signals	• DSC signal (total) • DSC signal (reversing) • DSC signal (non-reversing) • Specific heat (total) • Specific heat (reversing) • Specific heat (non-reversing) • Phase of specific heat • Specific heat (real part) • Specific heat (imaginary part)

Note: LabSolutions TA is required to use the TM-DSC program. TA-60WS is not compatible.

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Shimadzu Corporation

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