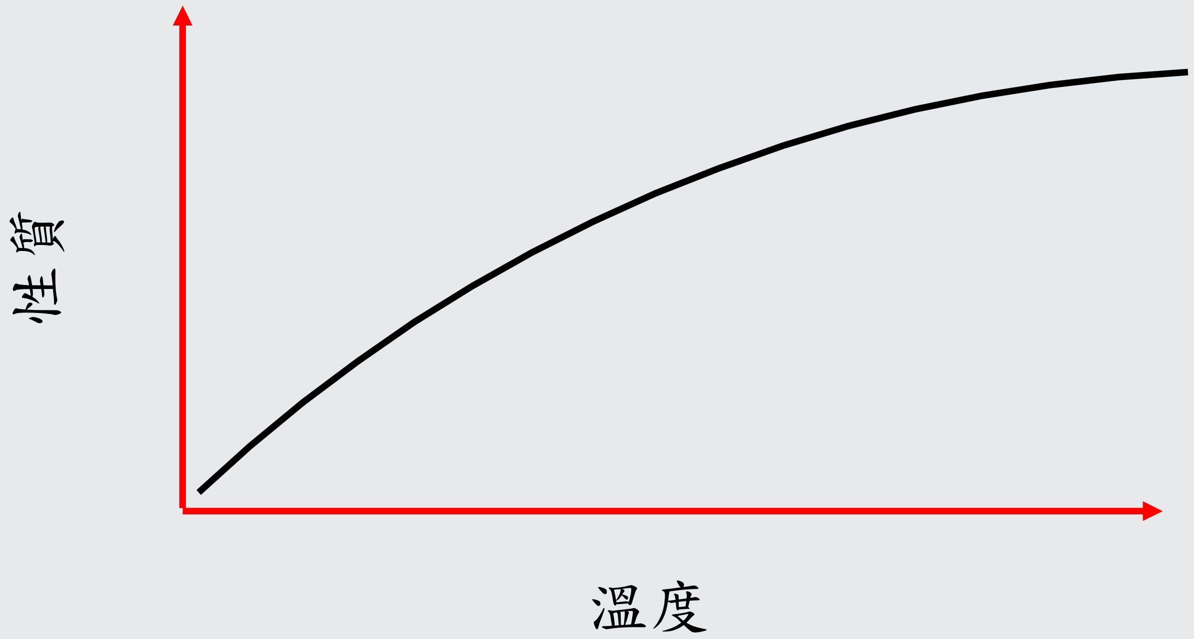
# DSC 204 ***F1*** *Phoenix* ®

# Features and Capability

# 熱 分 析

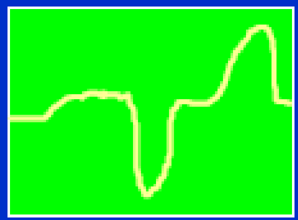
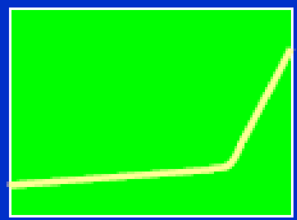
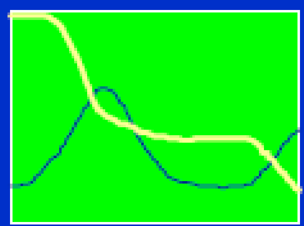
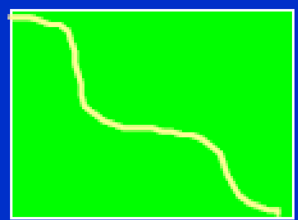
搜集物質的特性隨著溫度變化所相應的函數關係的技術稱為“熱分析”.



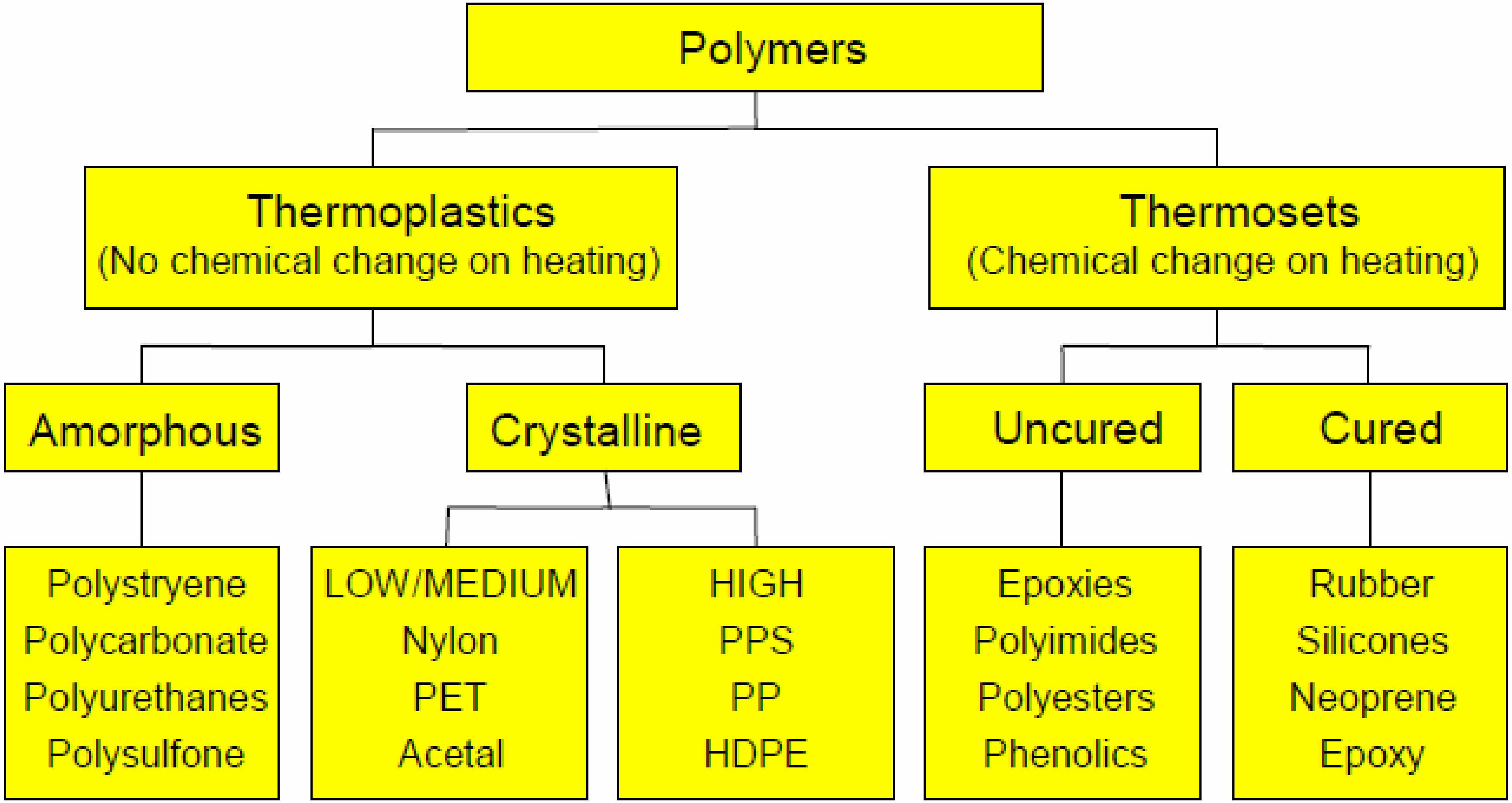
# 熱分析技術

- 示差掃描量熱儀(Differential Scanning Calorimetry DSC)
- 示差熱分析儀(Differential Thermal Analysis,DTA)
- 熱重分析儀(Thermogravimetric Analysis,TGA)
- 高解析度熱分析儀(High-Resolution TGA™)
- 示差掃描量熱-熱重分析聯用(Simultaneous DSC-TGA)
- 熱機械分析儀(Thermomechanical Analysis TMA)
- 動態熱機械分析儀(Dynamic Mechanical Analysis DMA)
- 介電性質分析(Dielectric Analysis DEA)
- 微熱分析儀-原子力顯微鏡與熱分析技術聯用- uTA (Micro TA - Atomic Force Microscope & TA)™

# *Thermal Analysis Techniques*

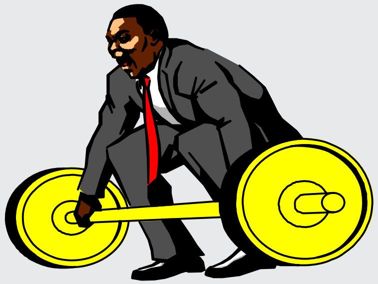
| | | |
|---|---|---|
| **DSC** |  | **Heat flow vs. Temp**<br>- Tm, Tc, Tg<br>- ΔH, curing time, curing degree<br>- Reaction rate, kinetics |
| **TMA** |  | **Dimensional Change vs. Temp**<br>- CTE<br>- Tg<br>- Softening point |
| **DMA** |  | **Viscoelastic property vs. Temp**<br>- Storage/Loss/Complex Modulus (E', E", E*)<br>- tan δ<br>- Viscosity and master curve |
| **TGA** |  | **Weight Loss vs. Temp**<br>- Decomposition temperature<br>- %Wt percentage<br>- Oxidative time |

# *Classifications of Polymers*

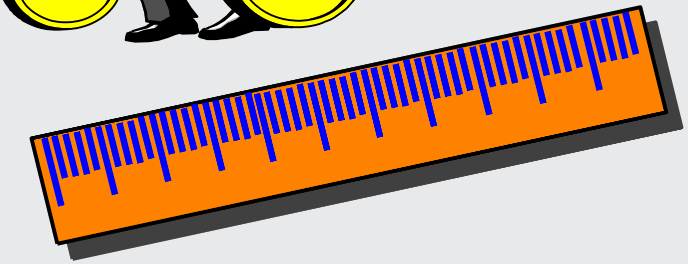


# 常用熱分析技術量測的物理特性

- 重量(Weight)-TGA



- 尺度(Length)-TMA



- 熱流(Heat Flow)-DSC

- 模量(Modulus)-DMA

# Differential Scanning Calorimeter

## （示 差 掃 描 量 熱 儀）

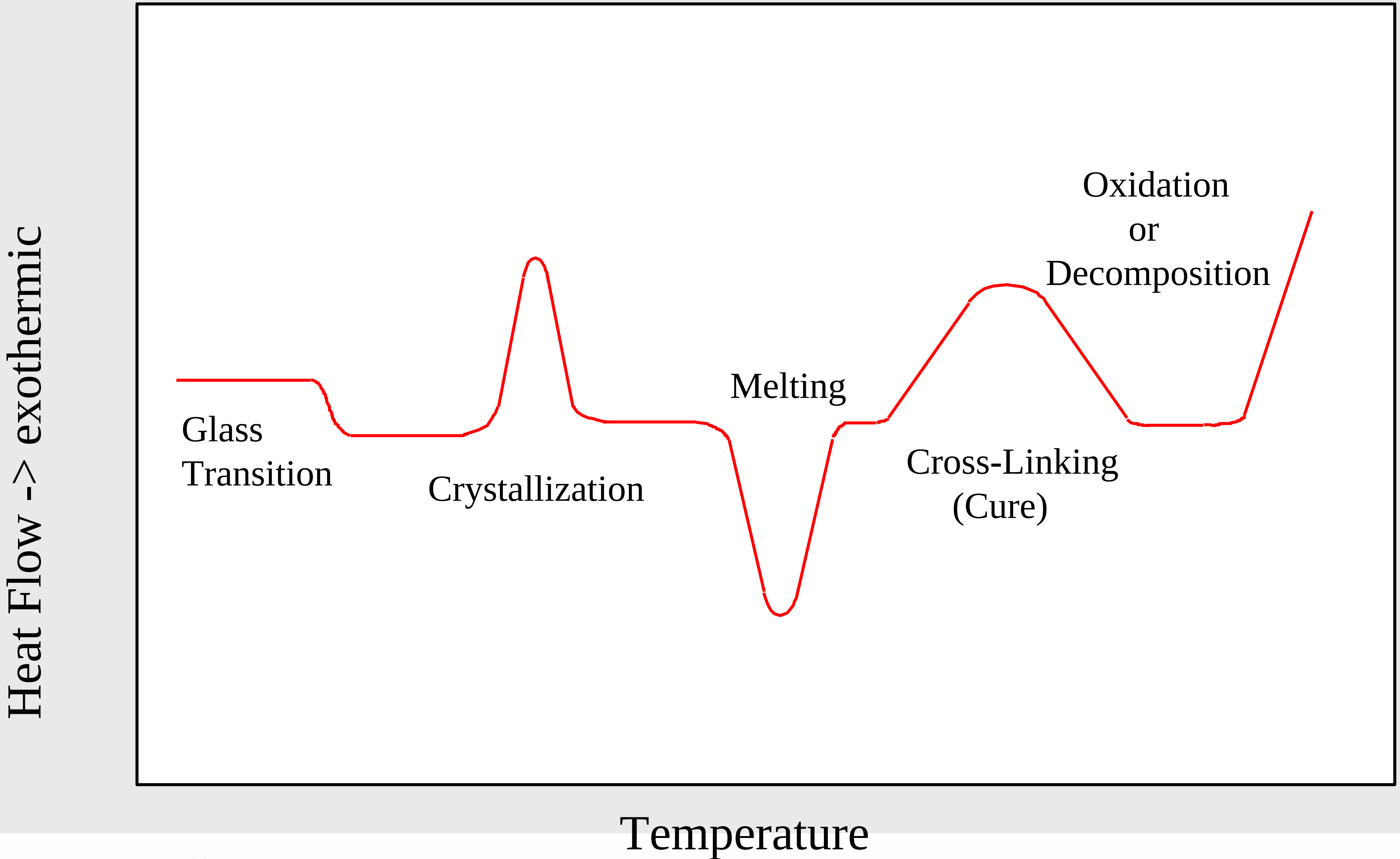
# (DSC)

在程式溫度（升／降／恒溫及其組合）過程中，測量樣品與參考物之間的熱流差，以表徵所有與熱效應有關的物理變化和化學變化。

# Applications of DSC

| | | | |
|---|---|---|---|
| 相變化點 | **Phase Transition** | 熔融熱 | **ΔH** |
| 玻璃轉移溫度 | **Tg** | 反應熱 | **ΔH** |
| 熔點 | **Melting point** | 活化能 | **Ea** |
| 冷結晶溫度 | **Crystal Temperature** | 氧化導引時間 | **O.I.T.** |
| 降溫結晶溫度 | **Cold Crystal Temperature** | 反應動力學 | **Dynamic** |
| 結晶度 | **Crystallinity** | 交連 | **Curing** |
| 結晶熱 | **Crystal Energy** | 純度 | **Purity** |
| 結晶半週期 | **Crystal Period** | 比熱 | **Cp** |

# DSC: Typical DSC Transitions

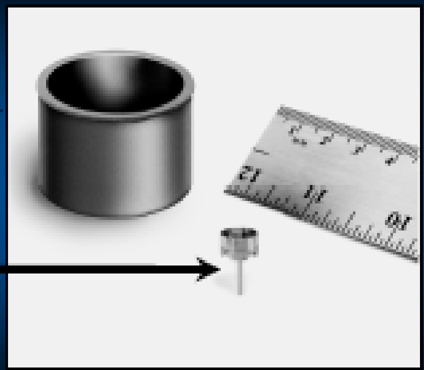


# The design principles of DSC

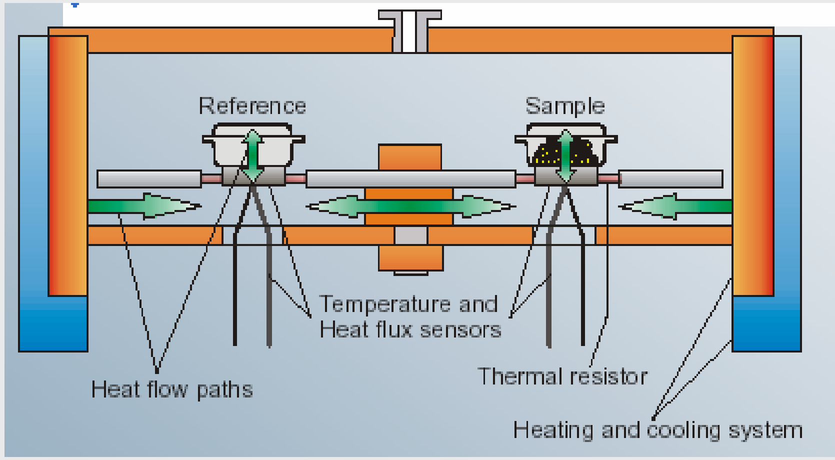
- ***Heat Flux DSC***
- ***Power Compensation DSC***

Heat Flux DSC furnace

Power Compensation DSC furnace



# *Heat Flux DSC function principle*

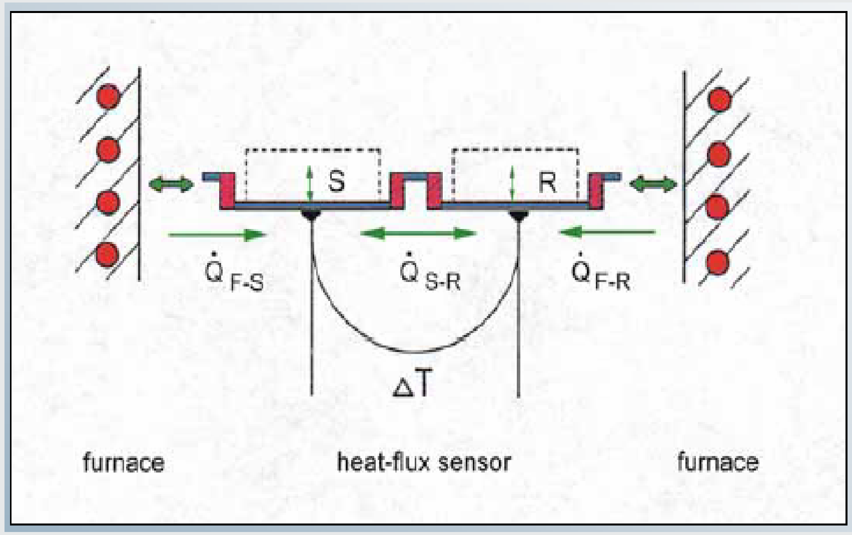


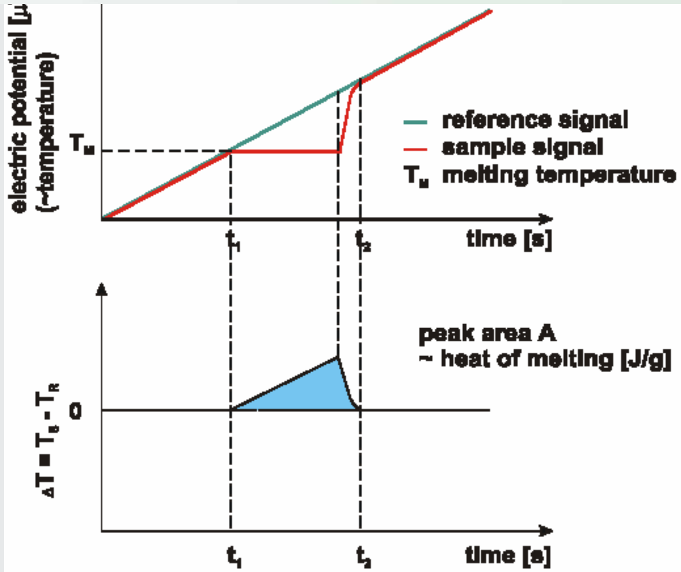

：樣品熱效應引起參比與樣品之間的熱流不平衡

$$Q = \lambda A \frac{\Delta T}{\Delta X}$$

：由於熱阻的存在，參比與樣品之間的溫度差（△T）與熱流差成一定的比例關係。將△T 對時間積分，可得到熱焓：

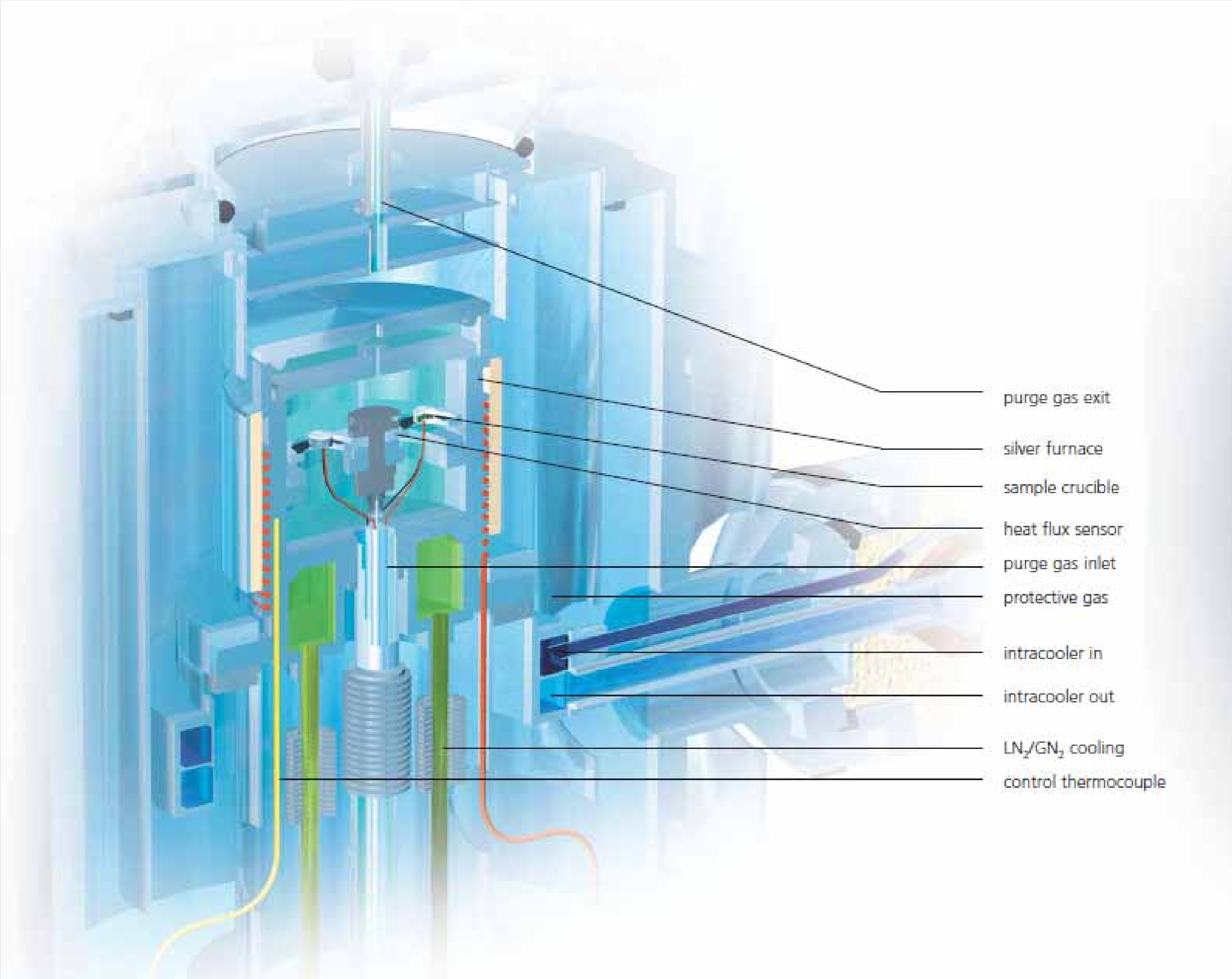
$$\Delta H = K \int_0^t \Delta T dt \qquad K = f\,(\text{溫度, 熱阻, 材料性質,...})$$



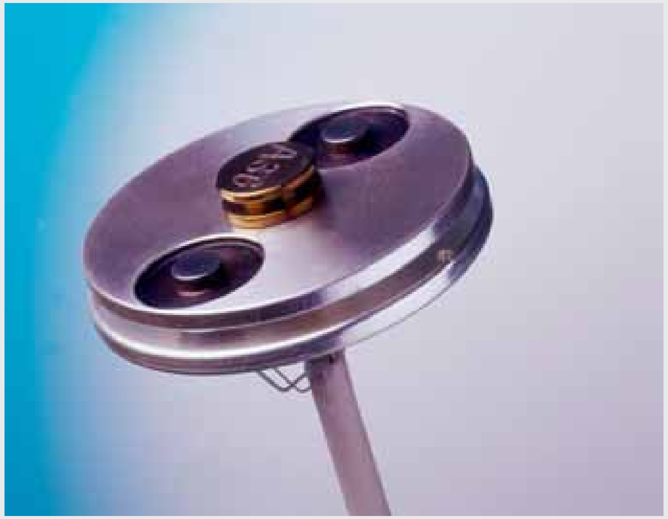




*DSC signal (first order transition)*

# DSC 204 *F1 Phoenix*®, Schematic Design



# Exchangeable DSC Sensor Types



## new τ–Sensor

***-180 ... 700℃***

***short response time***
***silver sensor***

## μ–Sensor

***-150 ... 400℃***

***high sensitivity***

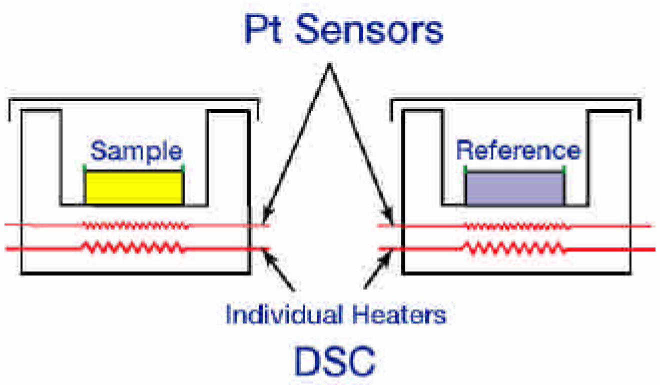
***silicon sensor***

τ – 感測器，回應速度最快，具有非常理想的峰分離能力

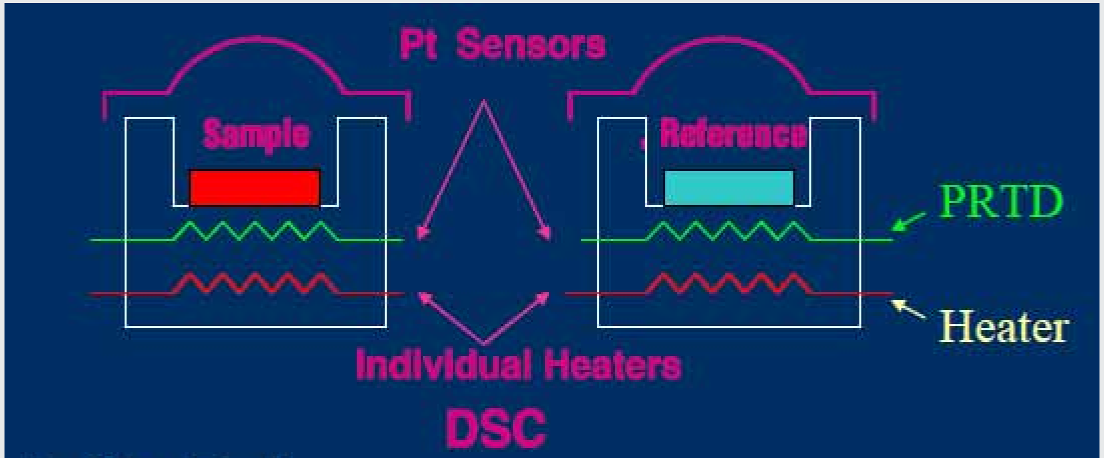
μ – 感測器，靈敏度爲普通感測器的十幾倍

# *Power Compensation DSC function principle*

熱補償式 Power Compensation DSC

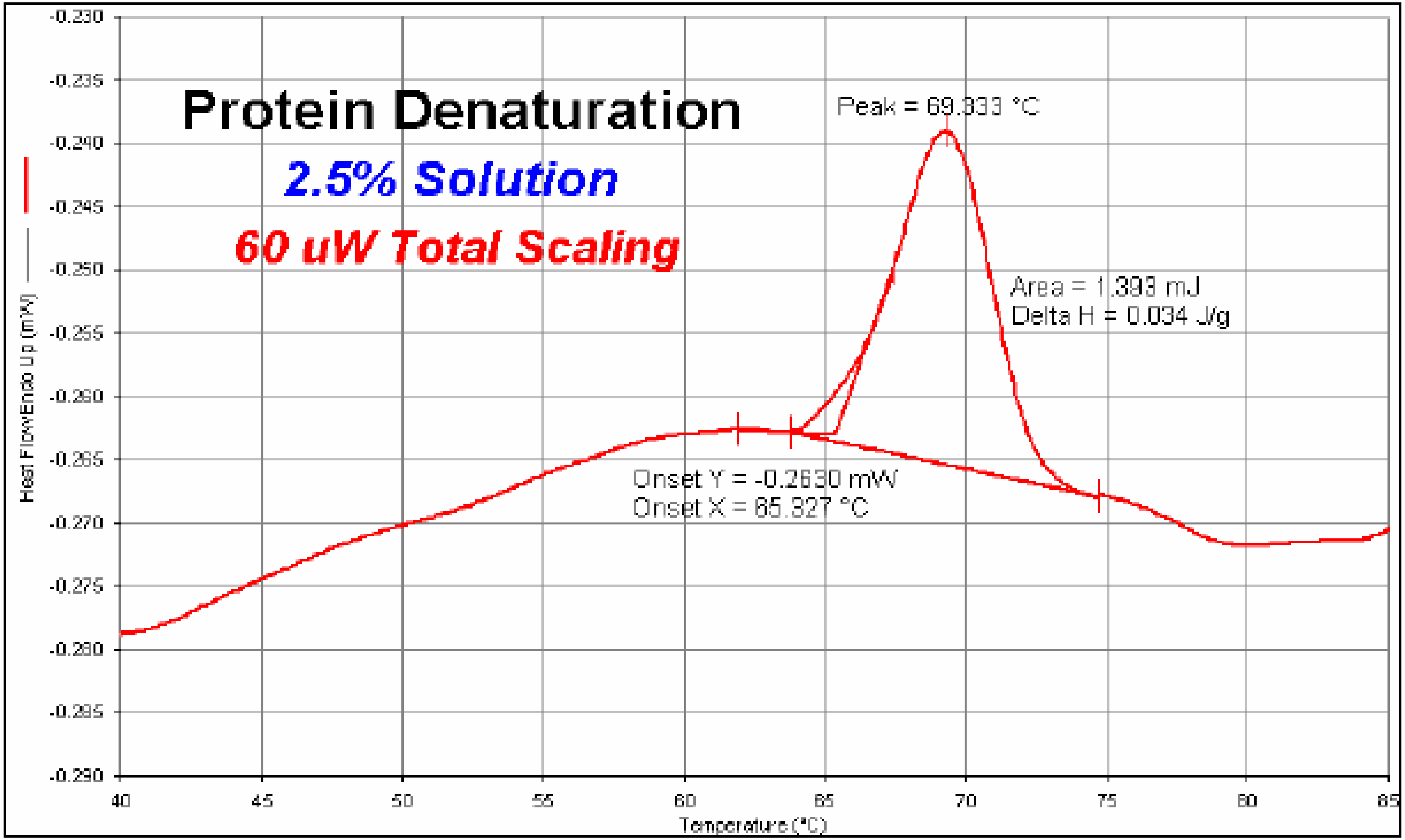



- -Measure ΔH directly
- - simple mathematical operations
- - smaller furnace

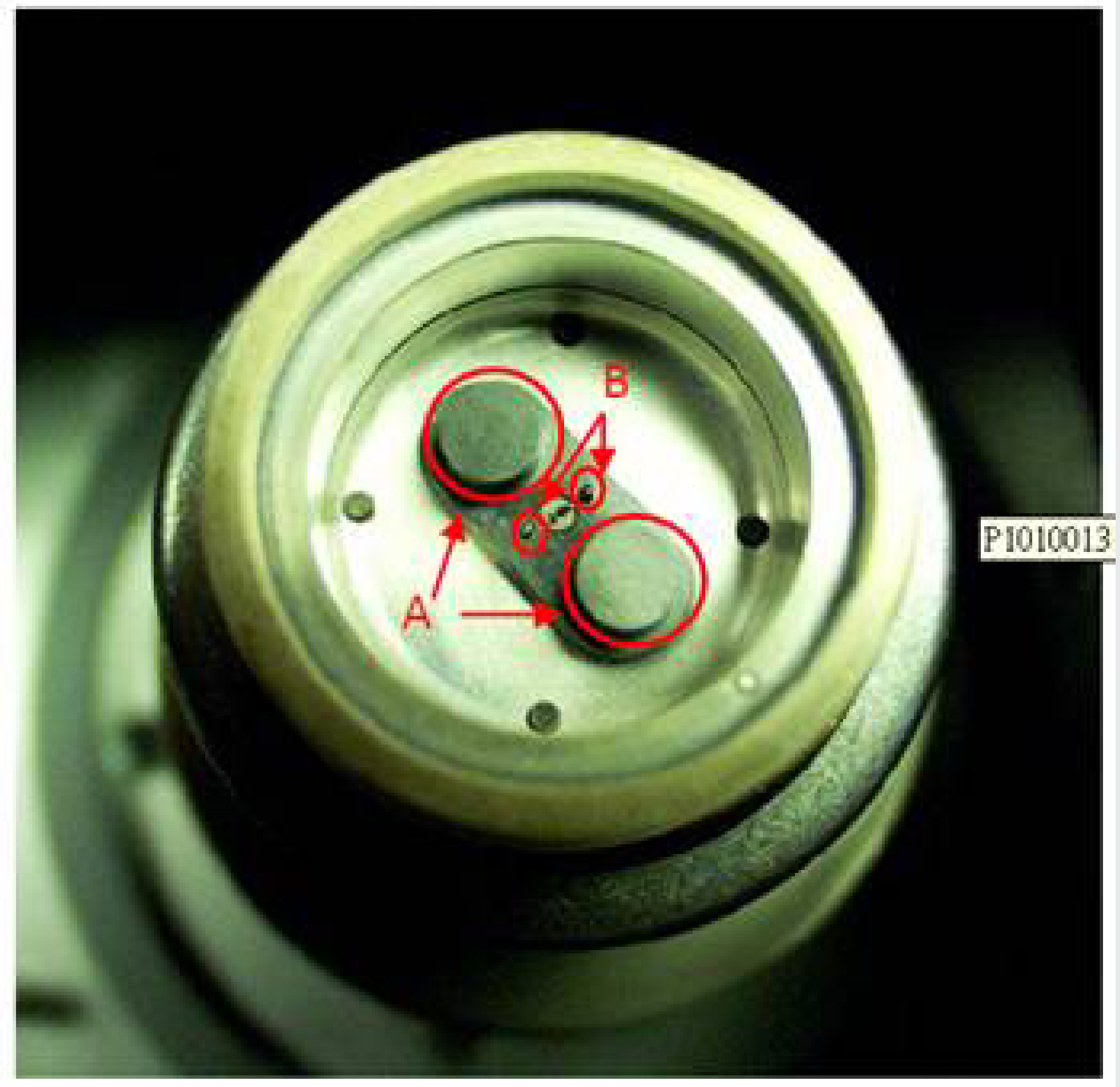



- Since $T_s = T_r$
- $\triangle H = (k)(\triangle W)$

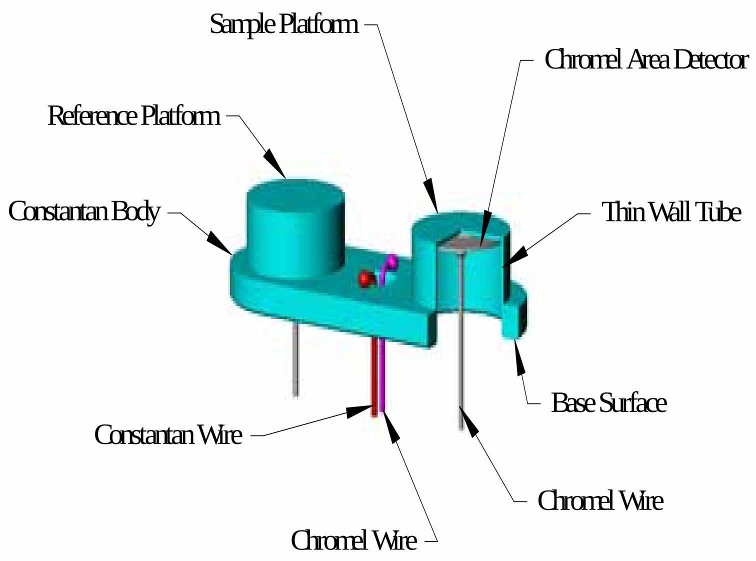
# *Protein Denaturation蛋白質變質測試*

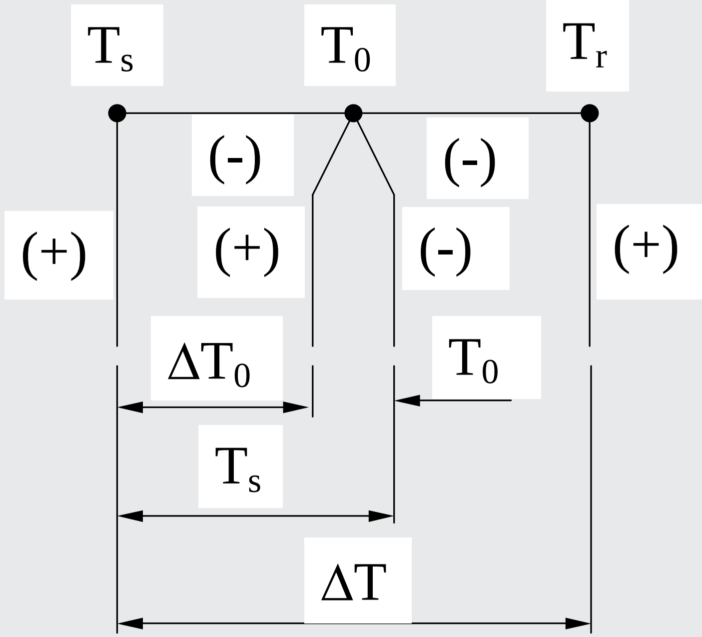


# TA 公司 - Tzero DSC Cell 構造



# Tzero Sensor Structure / Thermocouple Circuit




Constantan (-)

Chromel (+)

$T_0$ is used for temperature control

$$T_s = T_0 - \Delta T_0$$

# DSC 204 *F1 Phoenix*®, Schematic Design


***DSC 204 F1 Phoenix***®


***DSC 204 F1 Phoenix***® ***- ASC***

# DSC 204 F1 Auto Sampler

# DSC 204 *F1 Phoenix*®

NETZSCH

## Technical data

- Temperature range: -180 ... 700°C
- Heating and cooling rates: 0.001 ... 100 K/min
- DSC detection limit: 0.1 µW (depending on the sensor type)
- Exchangeable sensors: fast τ-sensor, high sensitive µ-sensor
- Several cooling devices: compressed air (to RT)
  Intracooler (-85 ... 600°C)
  liquid nitrogen (-180 ... 700°C)
- Electronically controlled gas flow for purge and protective gases
- Specific heat measurements possible (software option)
- Advaced Software available: Thermokinetics®, Peak Separation, Purity Determination, ChemReo®, etc.
- Automatic Sample Changer (ASC) for 64 samples and all crucibles

# DSC 204 F1 *Phoenix*®

## Already Prepared for Different Cooling Devices

***Liquid / gaseous nitrogen cooling***

***($LN_2$ / $GN_2$)***

***-180 ... 700℃***

Aluminium cooling body

***Intra-Cooler***

***-85 ... 600℃***

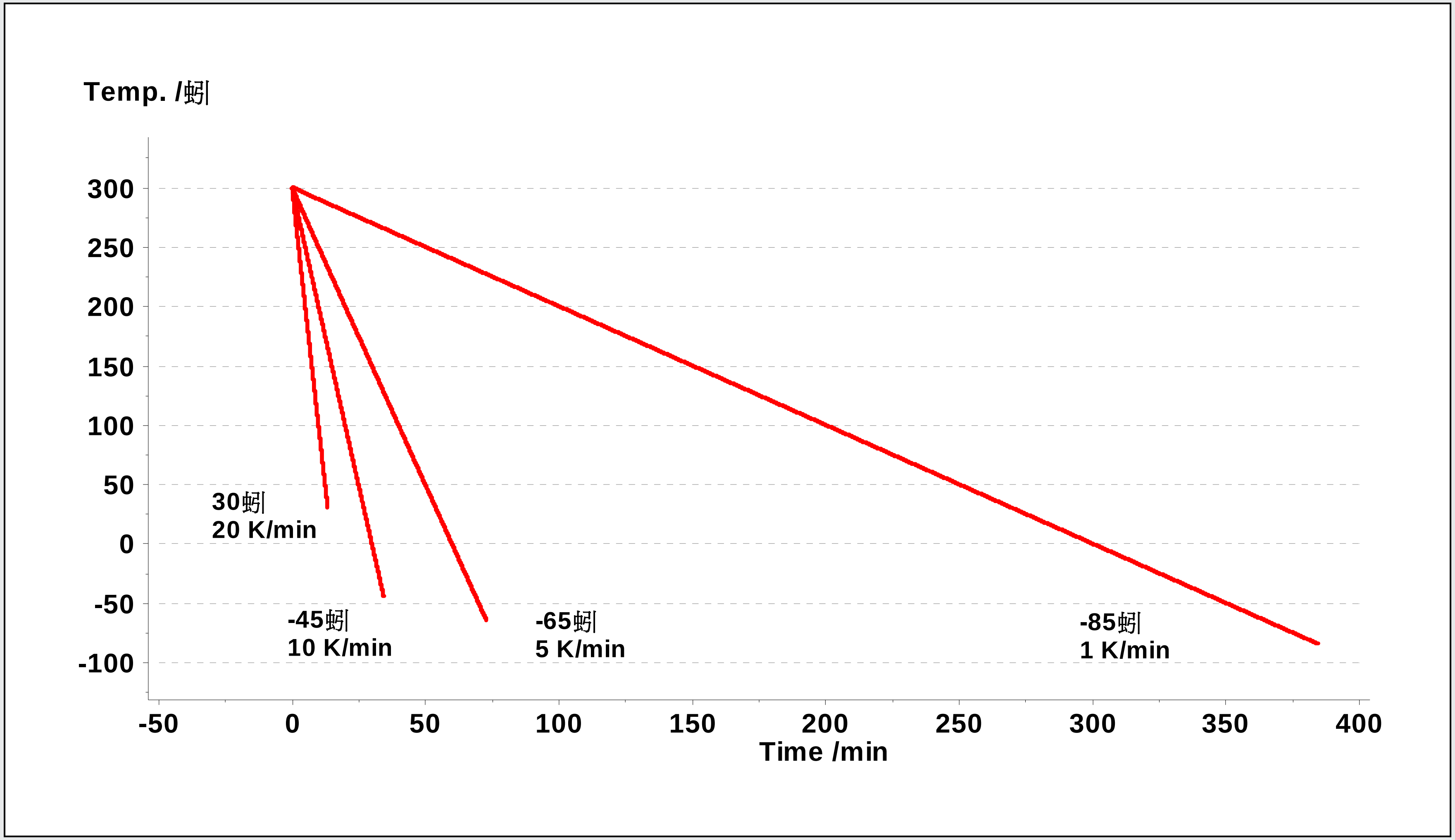
**Quench Cooling Can**


***Compressed air cooling***

***RT ... 700℃***

**Compressed air**

# DSC 204 *F1 Phoenix*®, Intracooler



# Netzsch DSC 的特點

NETZSCH

* 德國精密工藝製造;

*. 隱藏式爐體設計,不受外界環境影響.

* 熱流靈敏度高, 0.1 uW

* 基線穩定性, 10 uW

* 配置8點溫度校正, 溫度準確性高, 正確性 +/- 0.1°C, 精確性 +/- 0.01°C

C6H12 , Hg, KNO3, In, Bi, CsCl, Sn, Zn

* 爐體可長時間於 600°C 恆溫

* 爐體氣密性和熱絕緣性佳

* 整體成形的輕質銀鑄造, 不容易腐蝕, 使用壽命長

* 三路氣體設計, 保護爐體, 污染清除容易, 使用方便(選件MFC,質量流量控制)

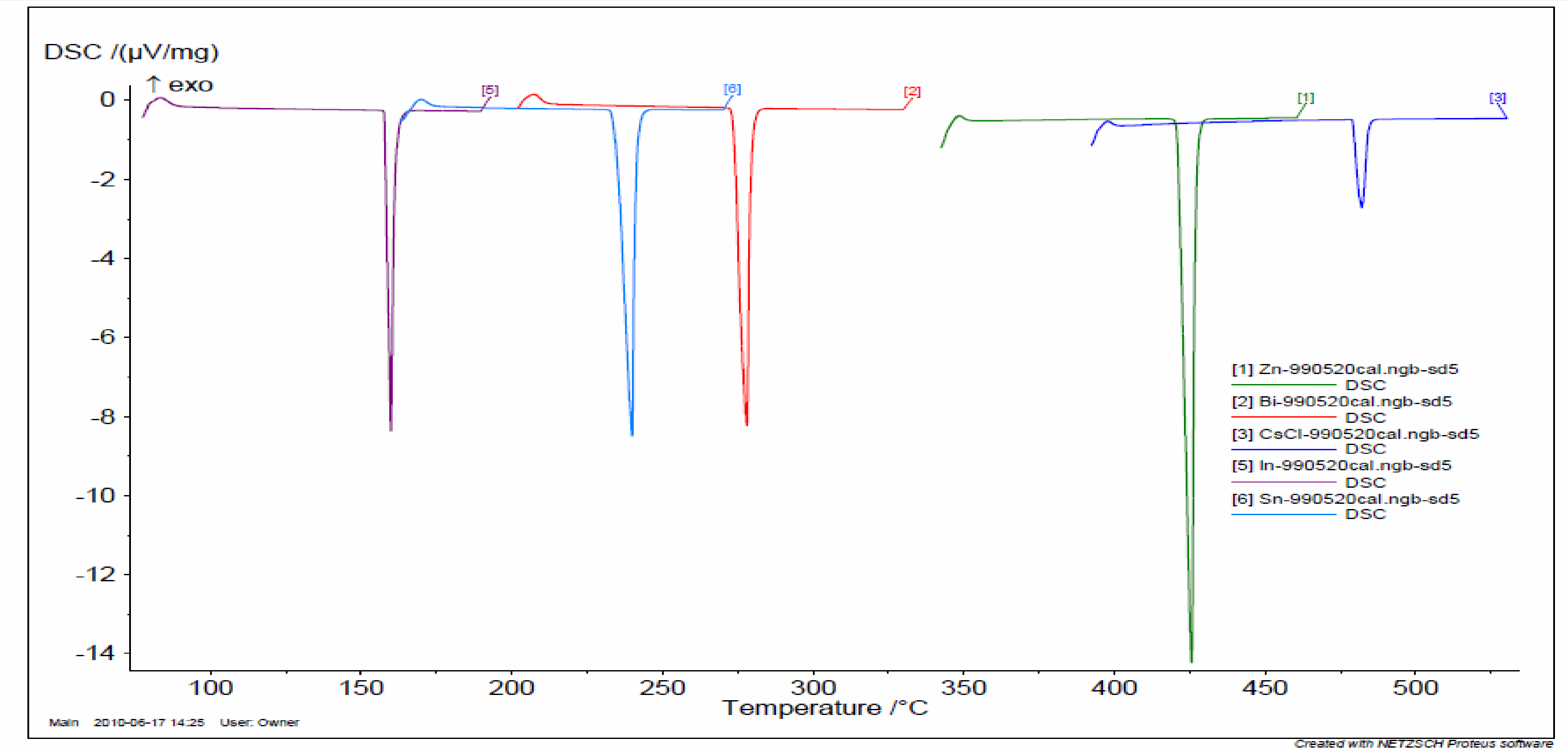
* 樣品自動進樣器(選配件)

*坩鍋多樣性: 固體, 液體, 高壓坩鍋, 等, 充分滿足需求

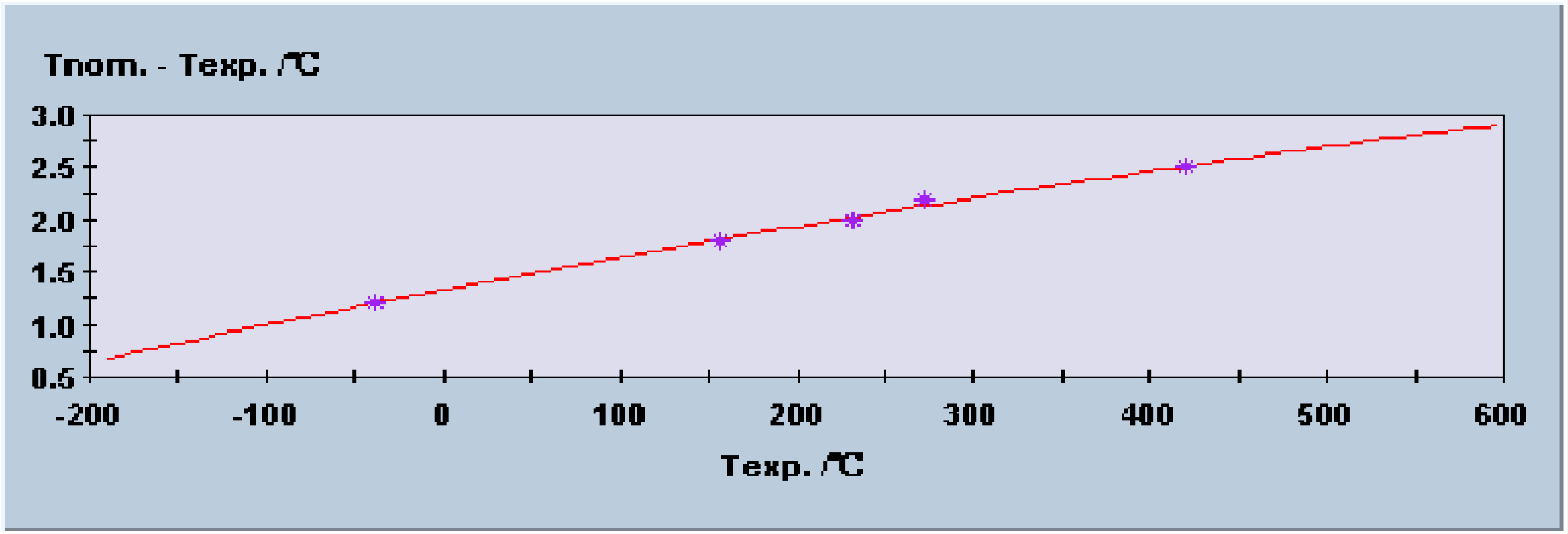
*因爲污染清除容易, 爐體不會腐蝕, 維修成本低廉

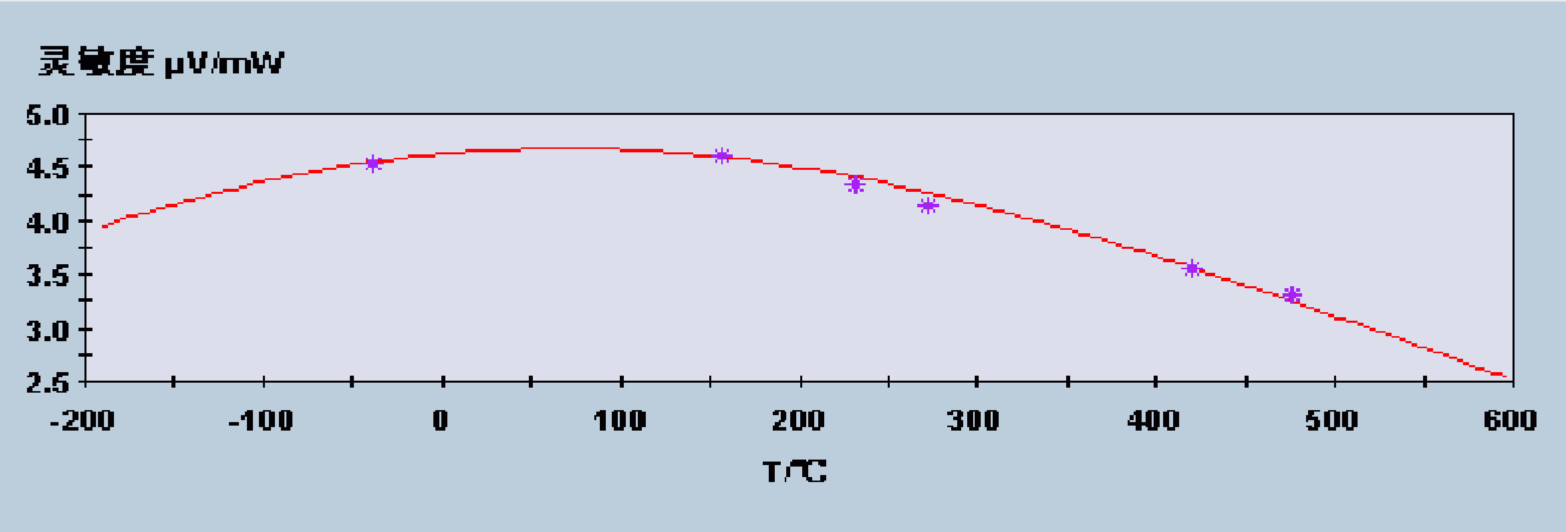
*DSC F1 真空度高, 可以和 FTIR 或 Mass 連用.

# DSC Calibration Curves

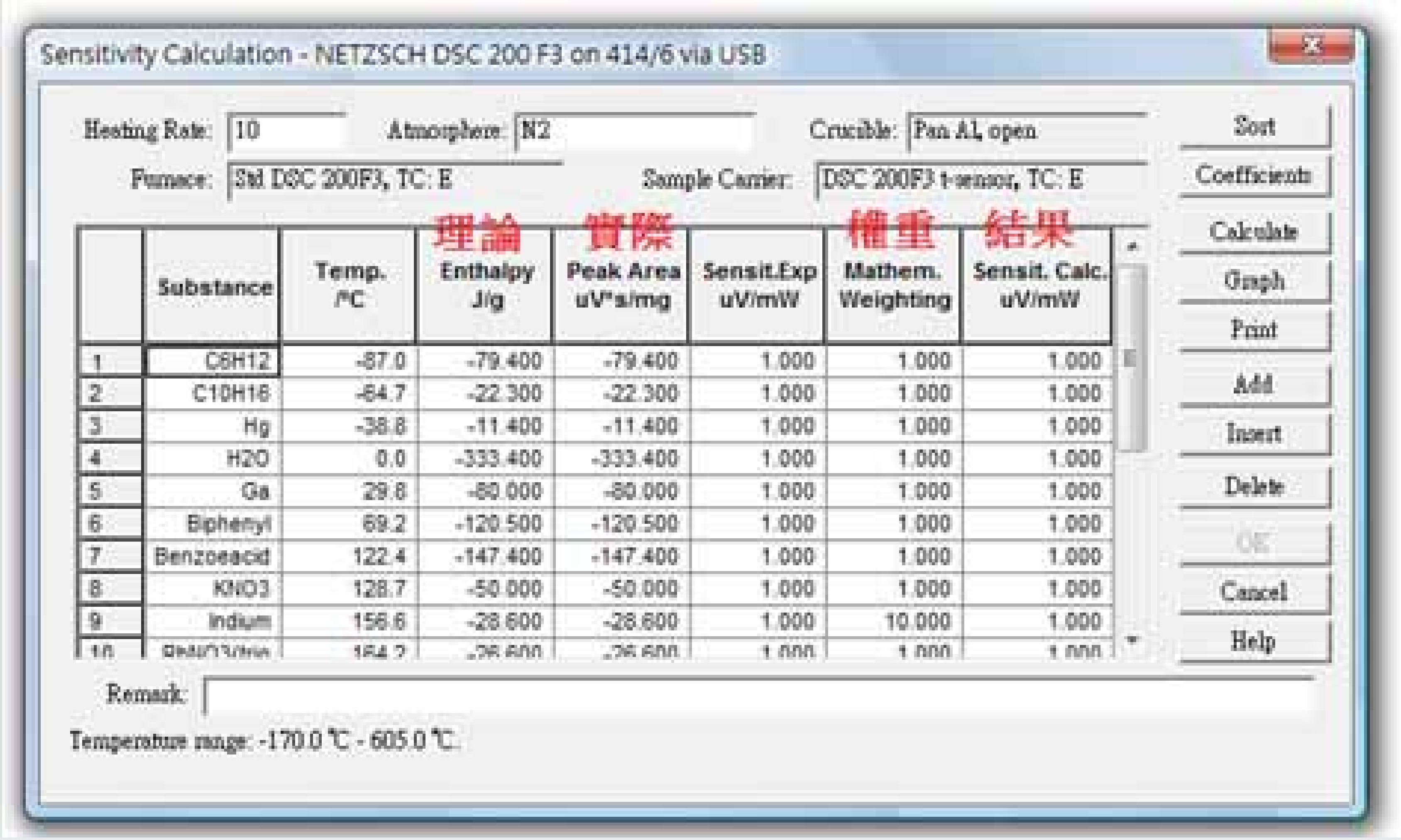


# DSC 溫度與熱焓的校正



# DSC 溫度與熱焓實際量測與理論值建檔

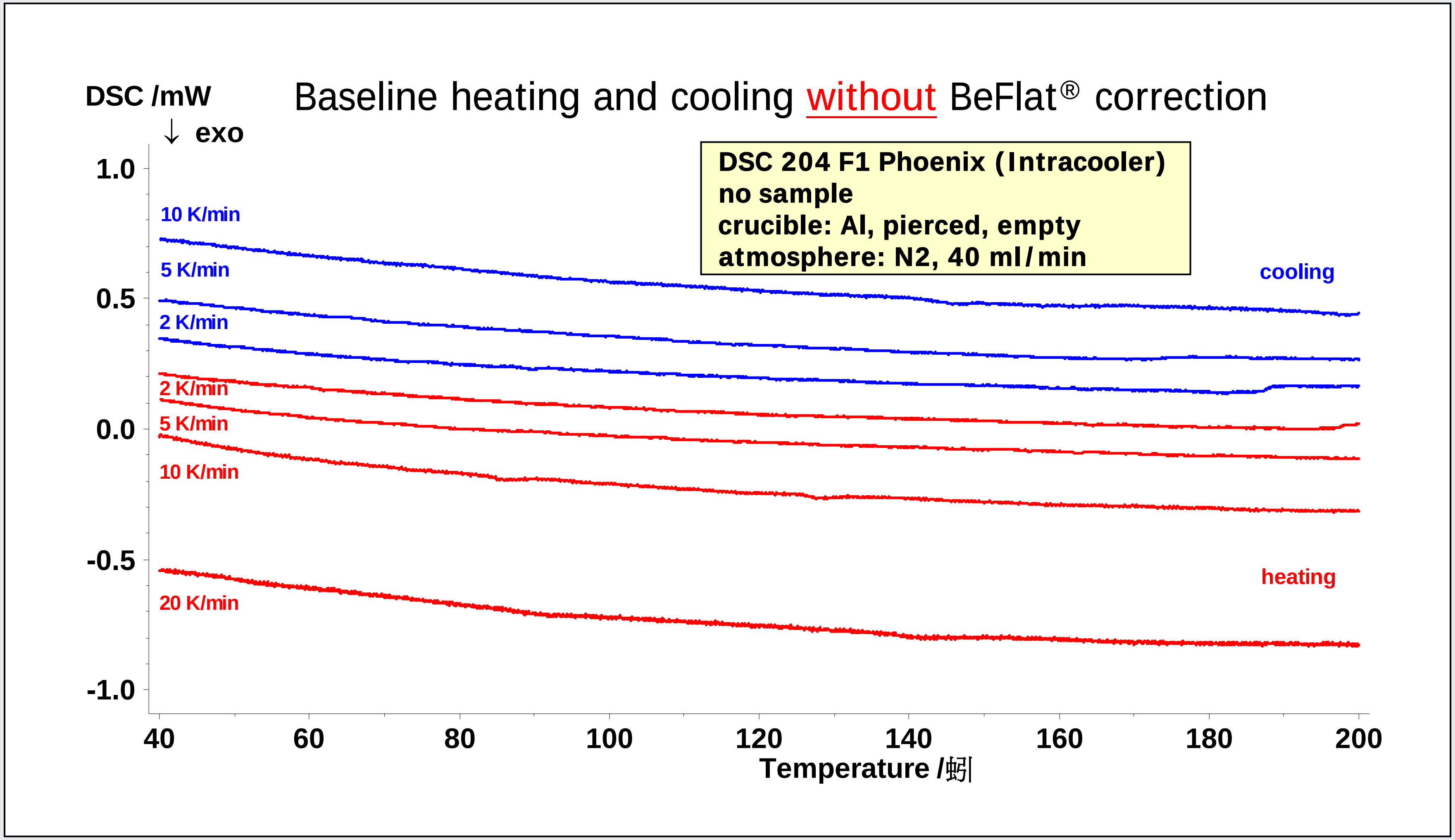



| | Substance | Temp. /°C | Enthalpy J/g | Peak Area uV*s/mg | Sensit.Exp uV/mW | Mathem. Weighting | Sensit. Calc. uV/mW |
|---|---|---|---|---|---|---|---|
| 1 | C6H12 | -87.0 | -79.400 | -79.400 | 1.000 | 1.000 | 1.000 |
| 2 | C10H16 | -64.7 | -22.300 | -22.300 | 1.000 | 1.000 | 1.000 |
| 3 | Hg | -38.8 | -11.400 | -11.400 | 1.000 | 1.000 | 1.000 |
| 4 | H2O | 0.0 | -333.400 | -333.400 | 1.000 | 1.000 | 1.000 |
| 5 | Ga | 29.8 | -80.000 | -80.000 | 1.000 | 1.000 | 1.000 |
| 6 | Biphenyl | 69.2 | -120.500 | -120.500 | 1.000 | 1.000 | 1.000 |
| 7 | Benzoeacid | 122.4 | -147.400 | -147.400 | 1.000 | 1.000 | 1.000 |
| 8 | KNO3 | 128.7 | -50.000 | -50.000 | 1.000 | 1.000 | 1.000 |
| 9 | Indium | 156.6 | -28.600 | -28.600 | 1.000 | 10.000 | 1.000 |
| 10 | RbNO3/trig | 164.2 | -26.600 | -26.600 | 1.000 | 1.000 | 1.000 |

# DSC 溫度與熱焓的理論值

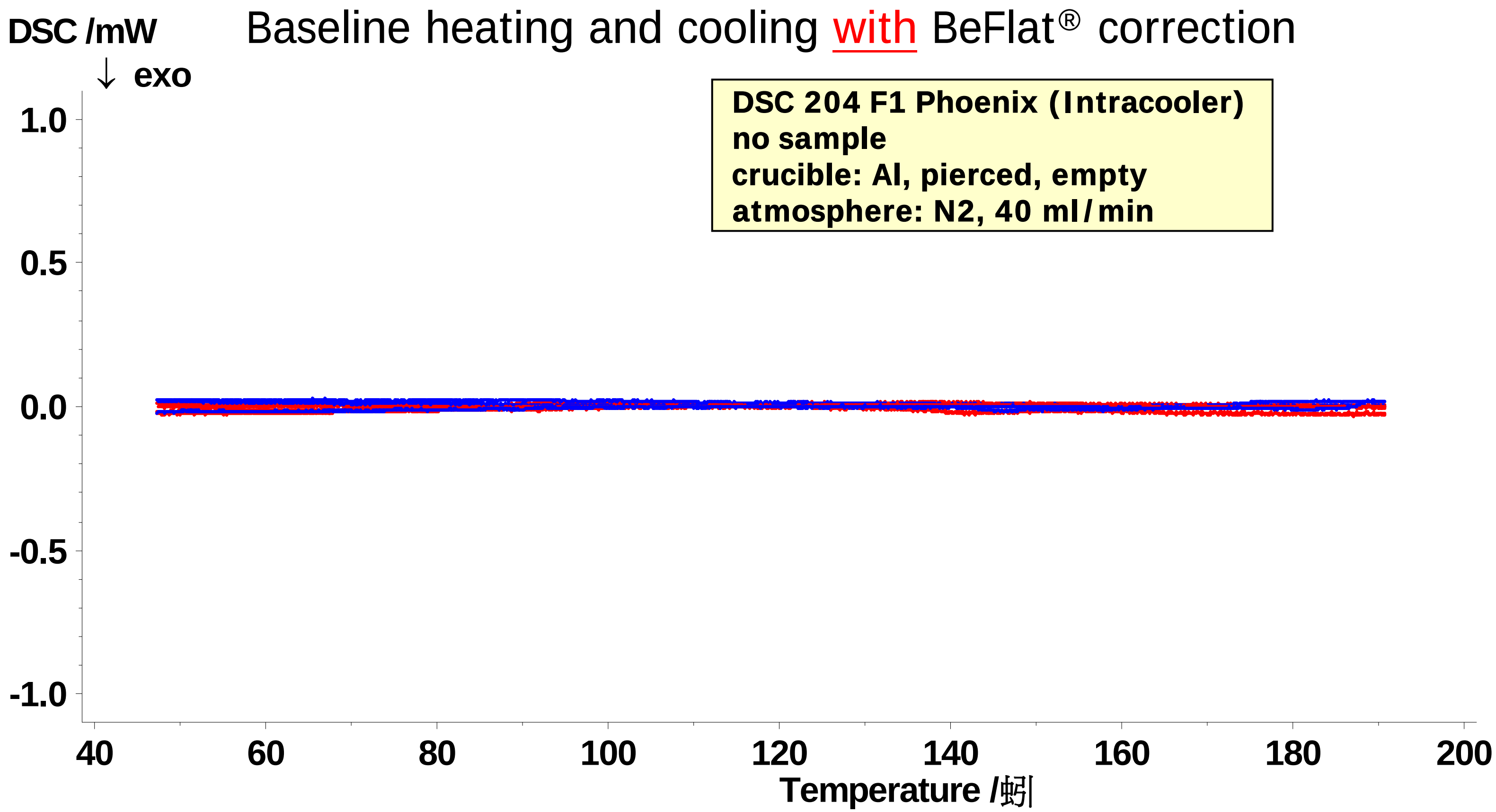
| 標準物質 | 理論熔點 ℃ | 理論熔融熱焓 J/g |
|---|---|---|
| C6H12 | -87 | -79.4 |
| Hg | -38.8 | -11.44 |
| KNO3 | 127.7 | -50.24 |
| In | 156.6 | -28.6 |
| Sn | 231.9 | -60.5 |
| Bi | 271.4 | -53.3 |
| Zn | 419.5 | -107.5 |
| CsCl | 476 | -17.22 |

# BeFlat



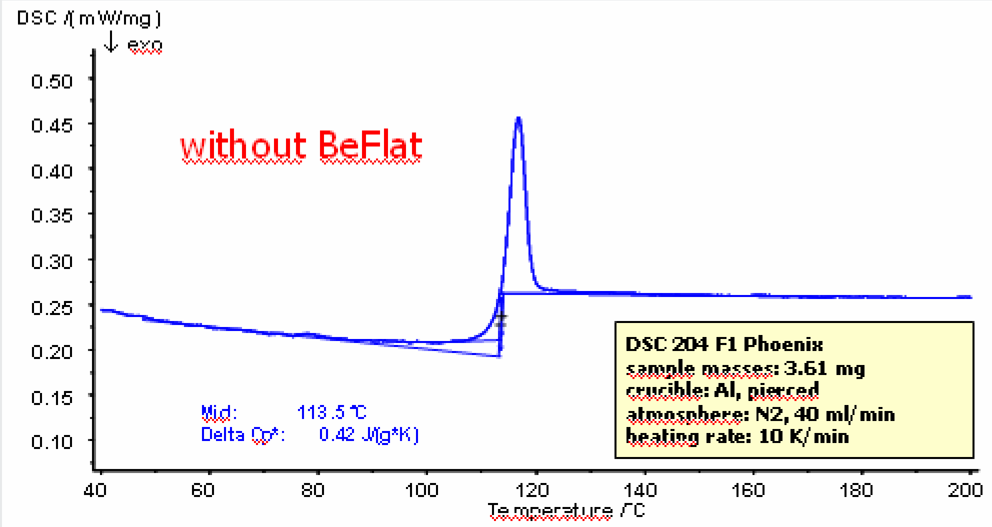
DSC /mW
↓ exo
Baseline heating and cooling without BeFlat® correction
DSC 204 F1 Phoenix (Intracooler)
no sample
crucible: Al, pierced, empty
atmosphere: N2, 40 ml/min
1.0
0.5
0.0
-0.5
-1.0
10 K/min
5 K/min
2 K/min
2 K/min
5 K/min
10 K/min
20 K/min
cooling
heating
40
60
80
100
120
140
160
180
200
Temperature /蚓

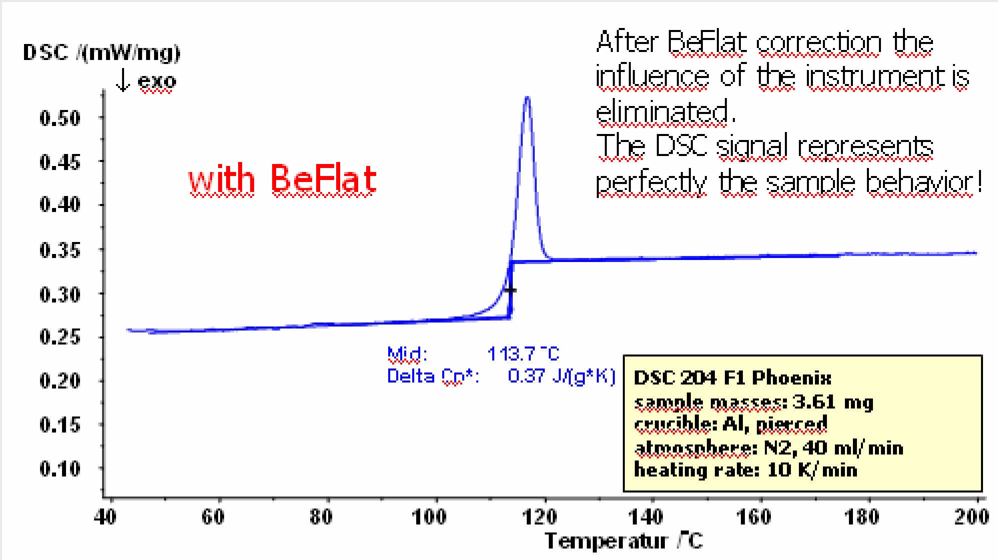
# BeFlat



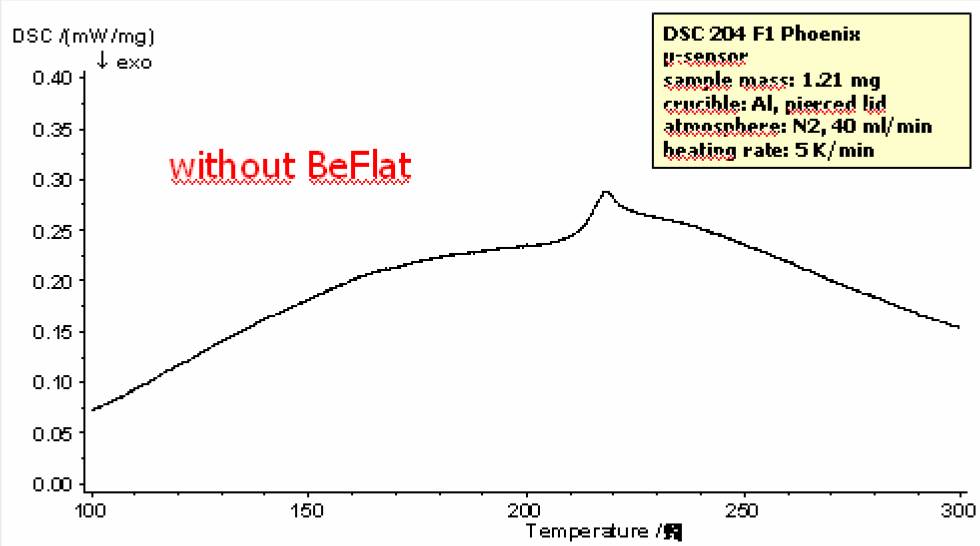
DSC /mW
↓ exo
Baseline heating and cooling with BeFlat® correction
DSC 204 F1 Phoenix (Intracooler)
no sample
crucible: Al, pierced, empty
atmosphere: N2, 40 ml/min
1.0
0.5
0.0
-0.5
-1.0
40
60
80
100
120
140
160
180
200
Temperature /蚓

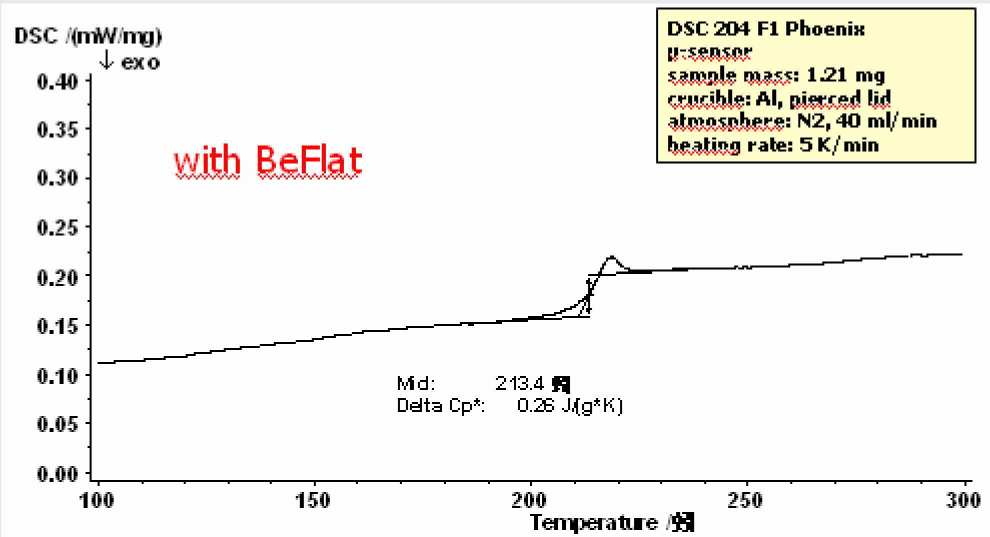
# BeFlat (Styrene-Acrylonitrile, SAN)



# BeFlat (Polyetherimide, PEI)

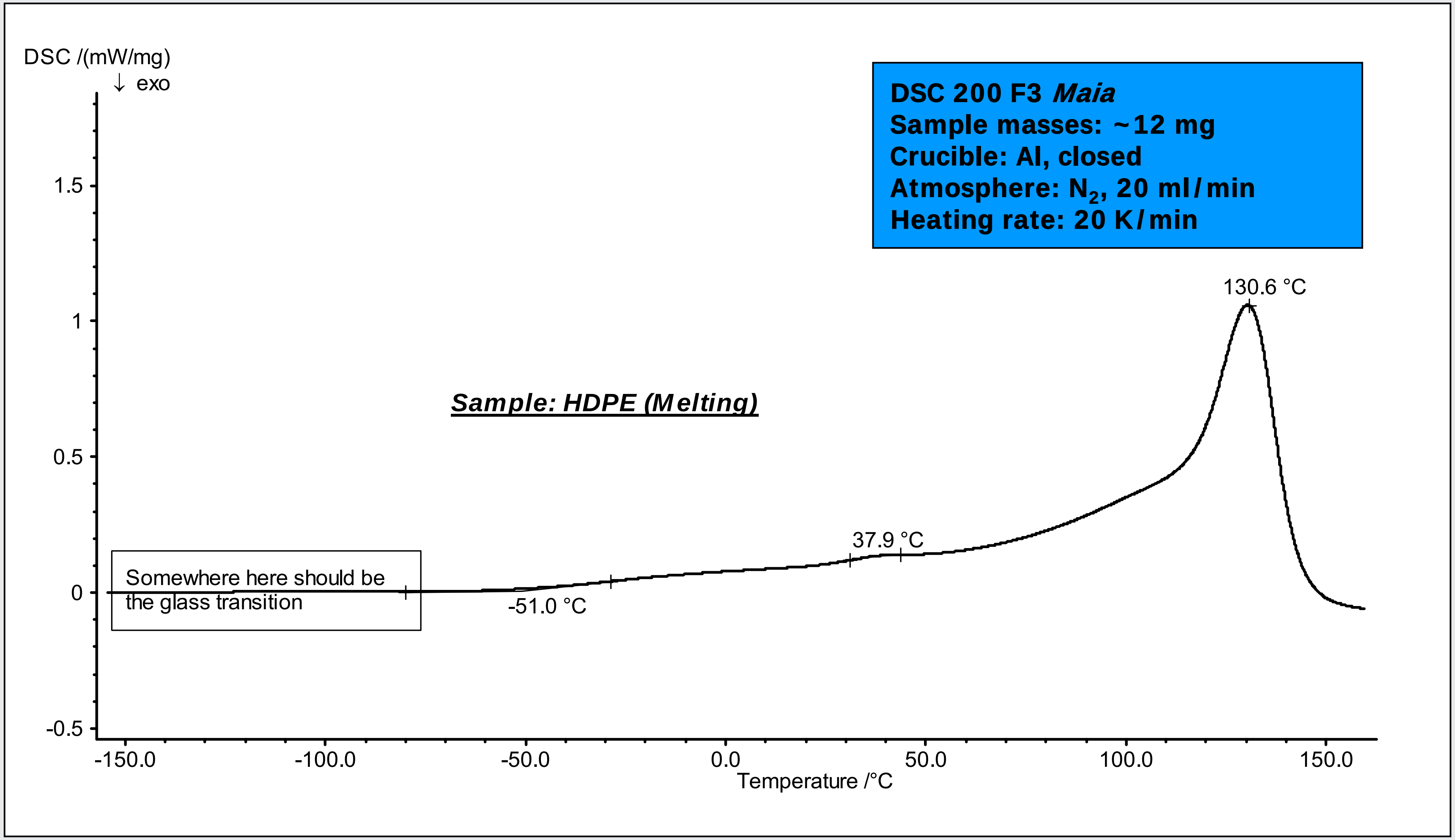


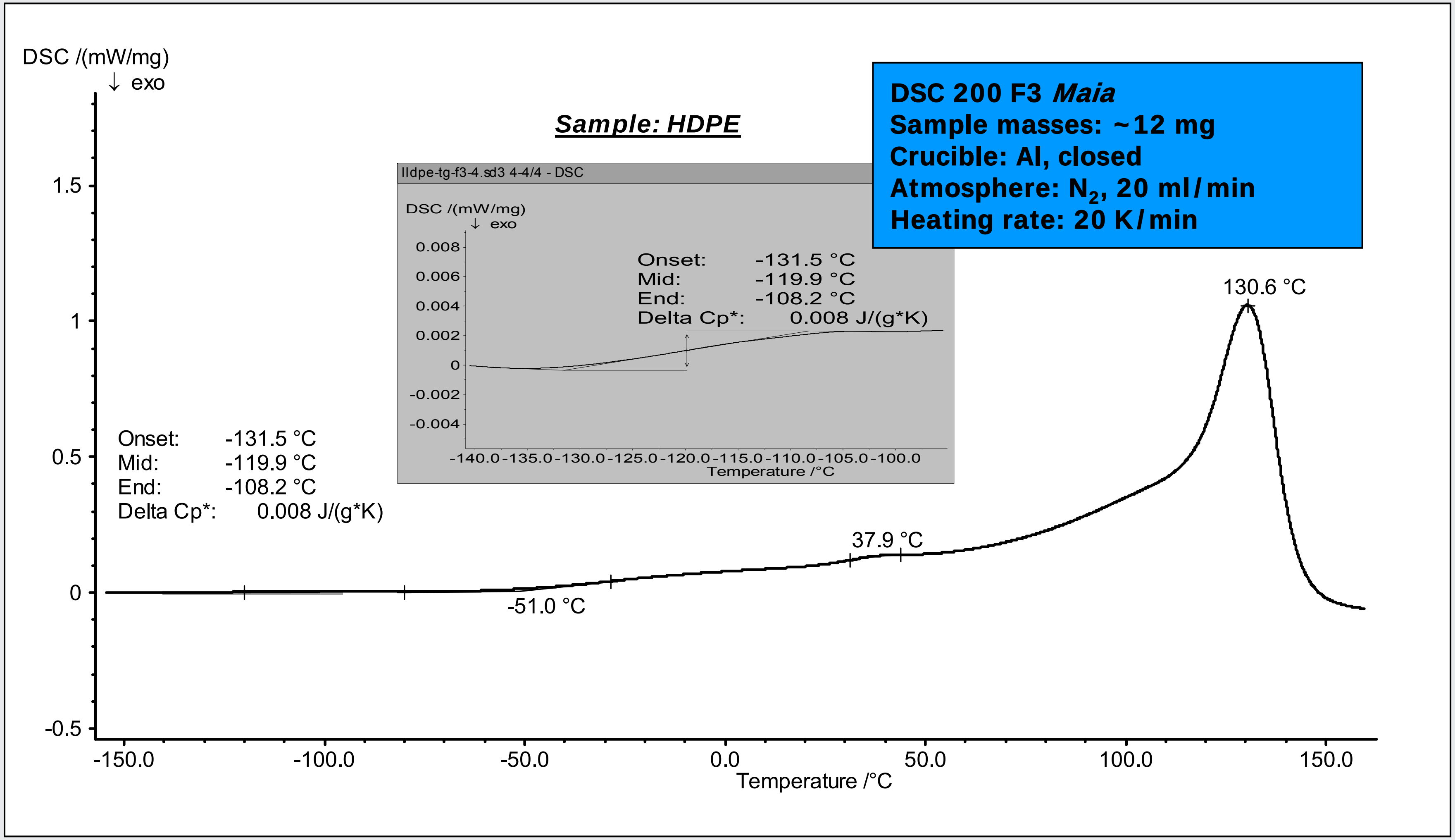
# Application – HDPE-Packaging

HDPE = High-Density Polyethylene

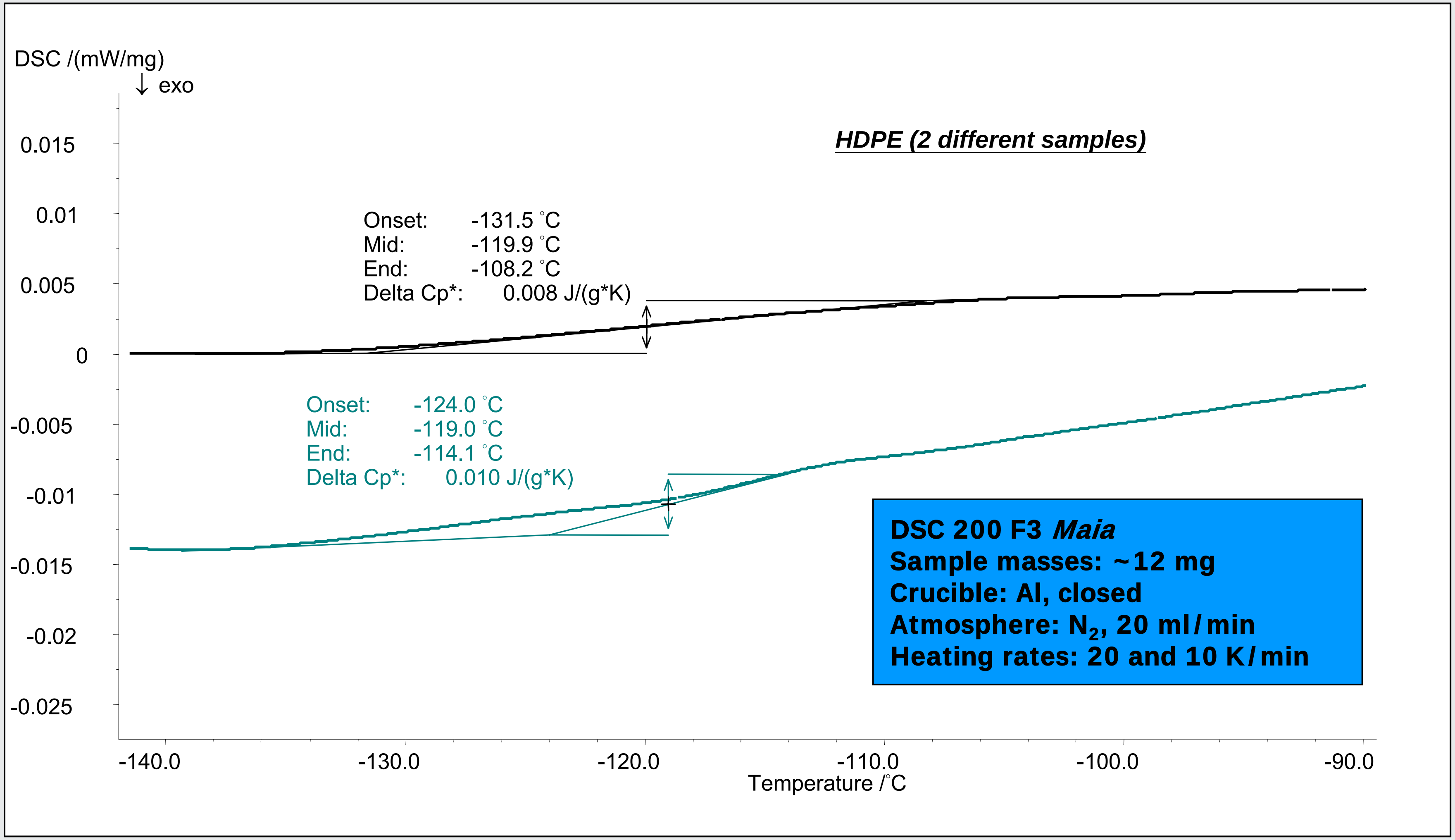

# Application - HDPE



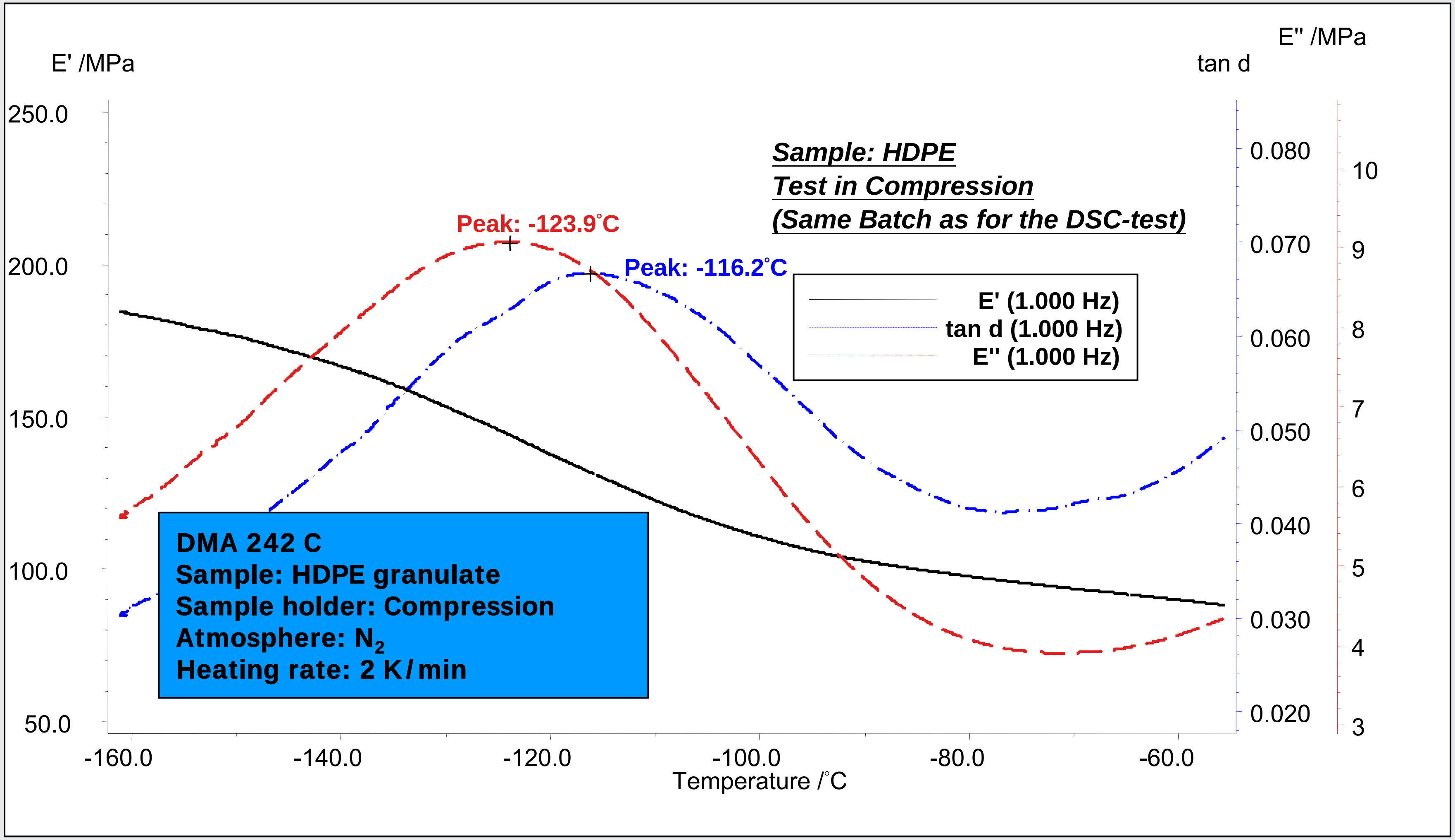
# Application - HDPE



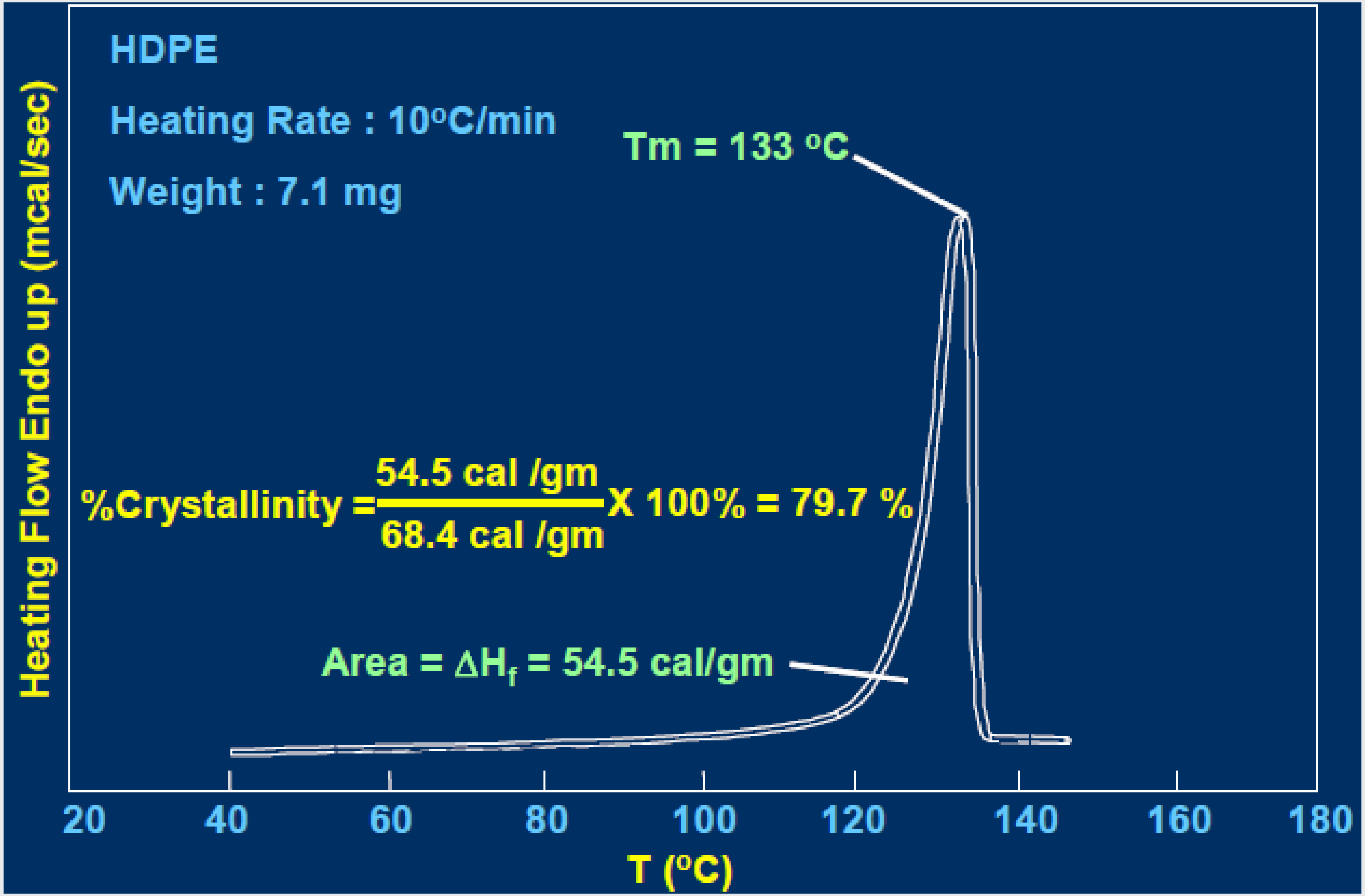
# Application - HDPE



# Application - HDPE

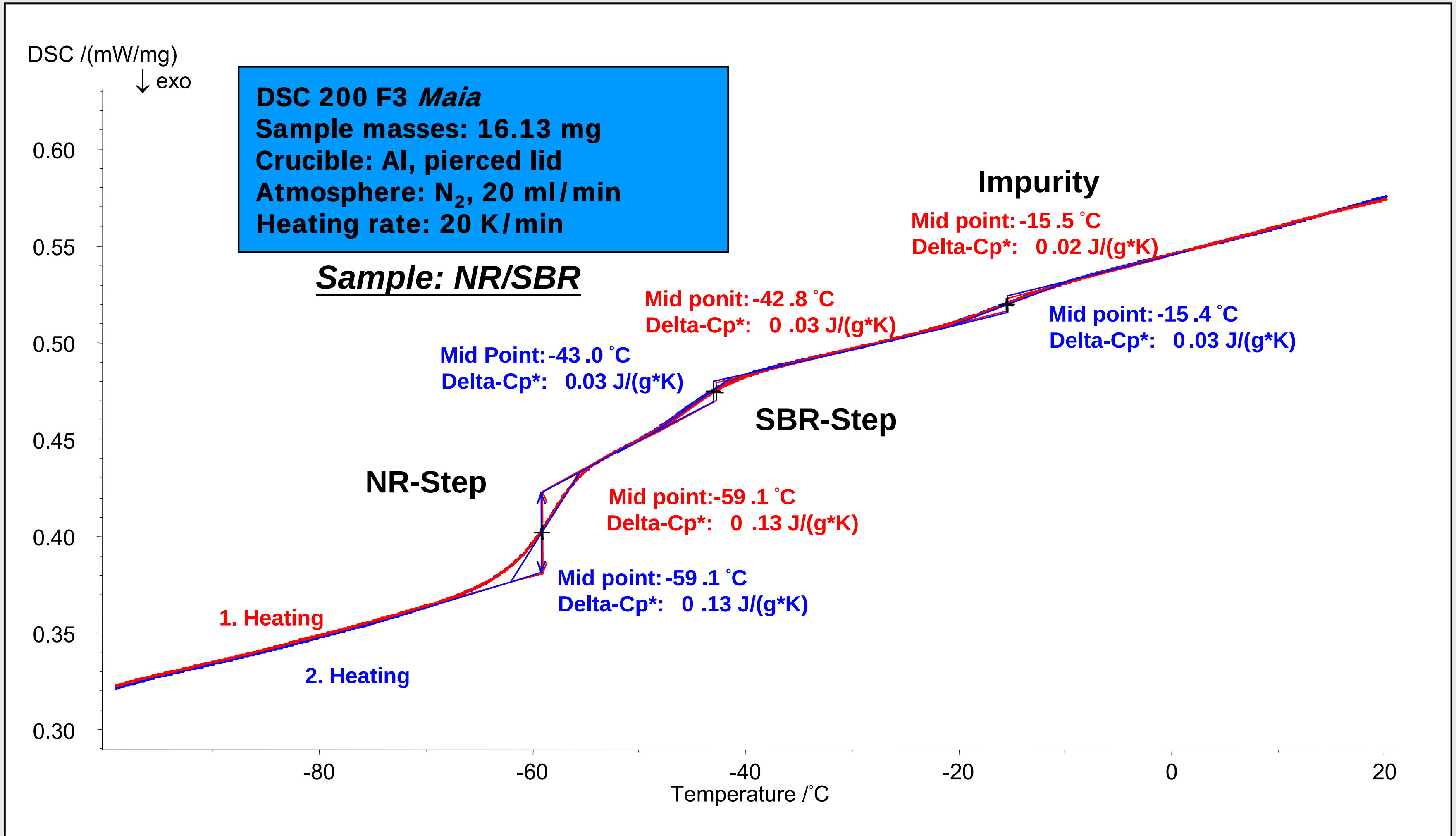


# *Analysis of Tm–calculation of crystallinity*



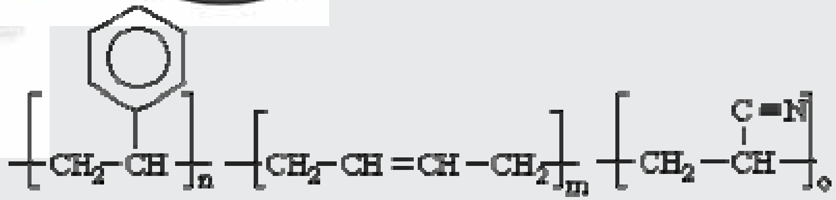
# Application – NR/SBR

# Application – NR/SBR

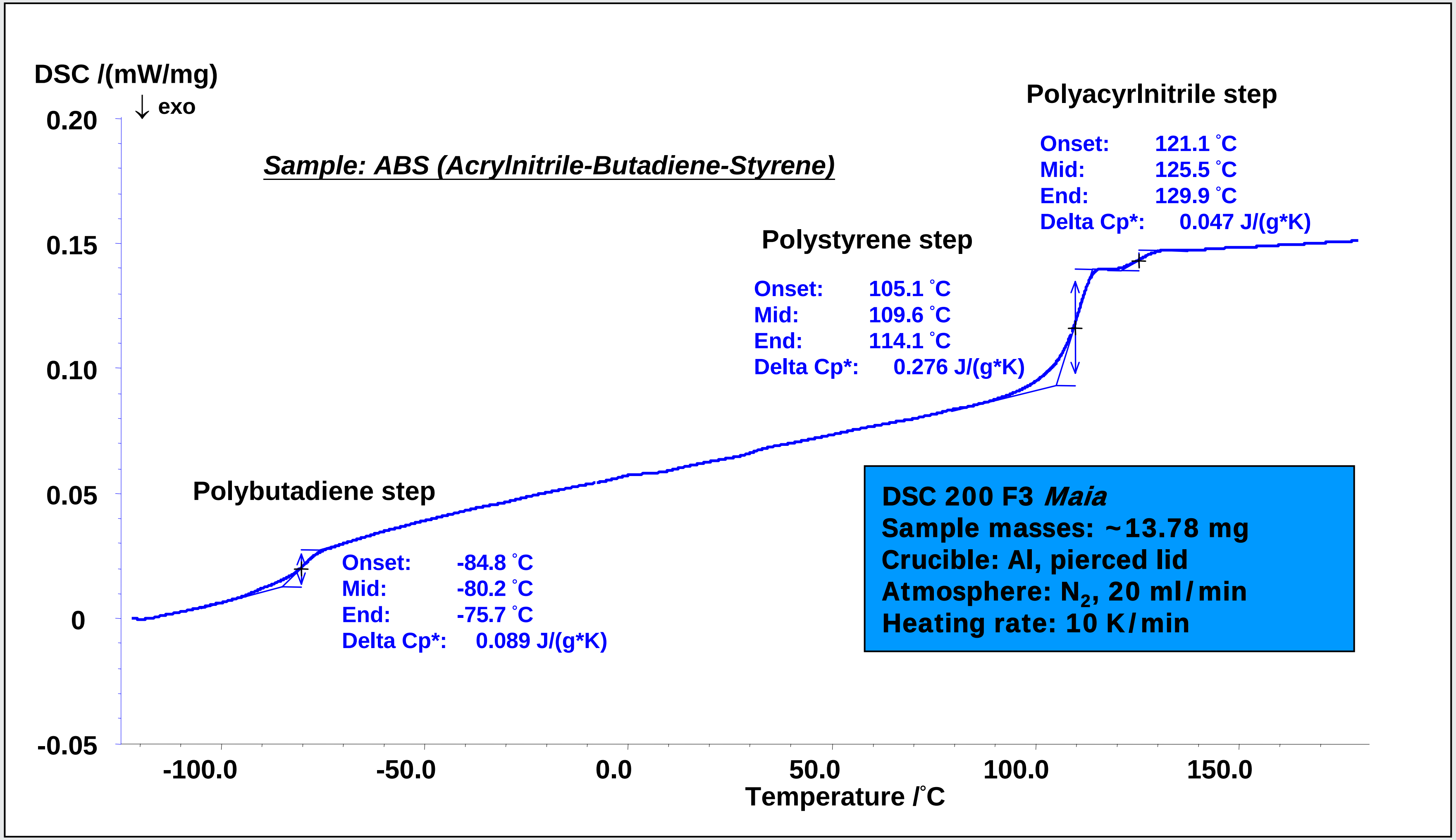


ABS = Acrylnitrile-Butadiene-Styrene-Terpolymer

# Application – ABS

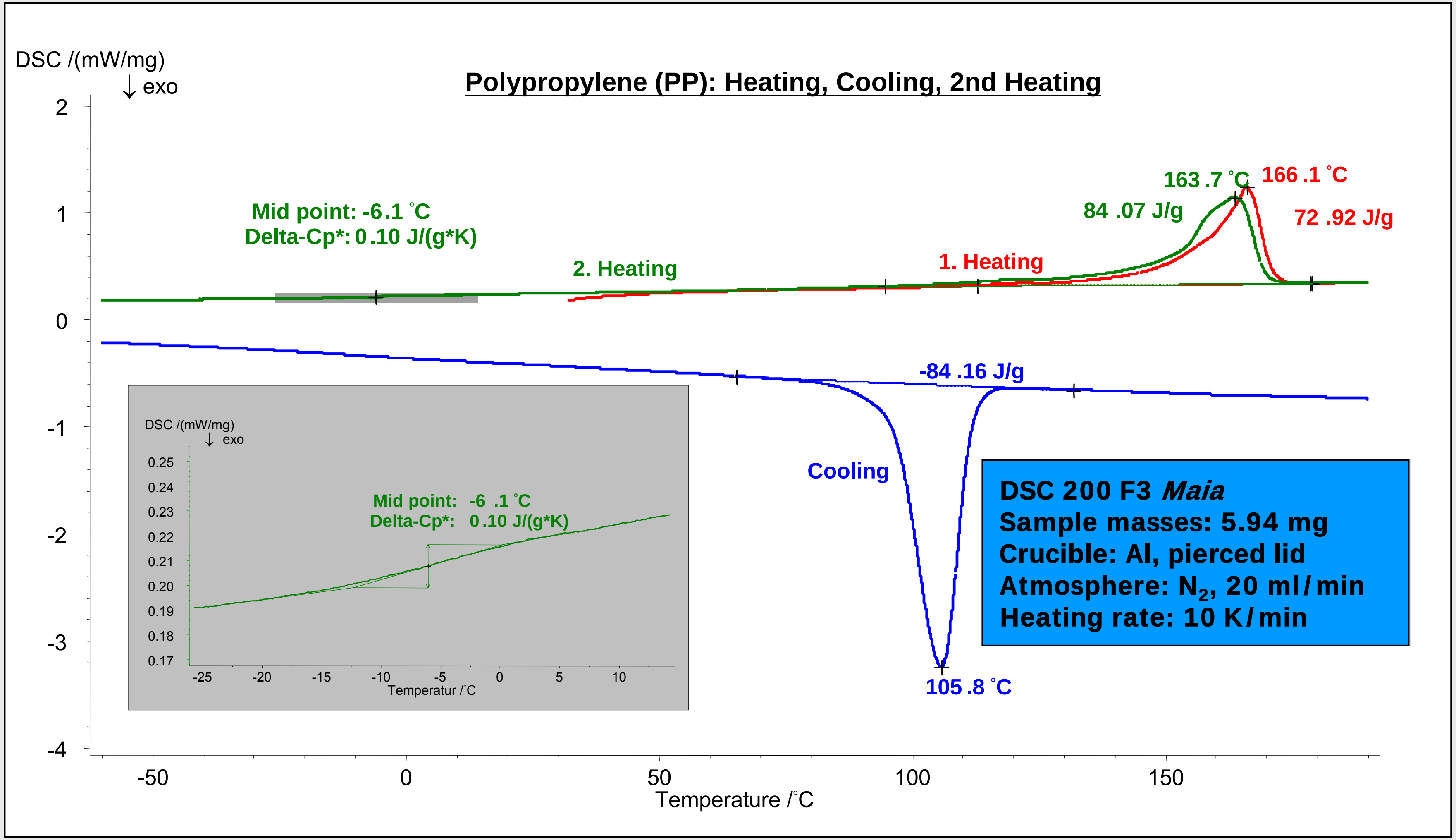



# Application – ABS

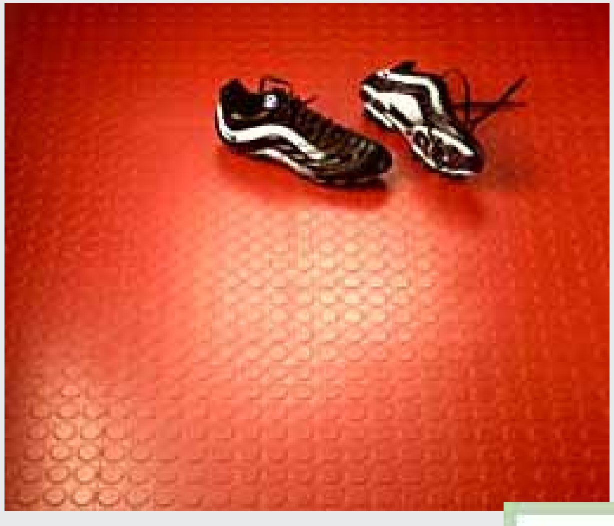


# PP = Poly-Propylene

# Application – PP

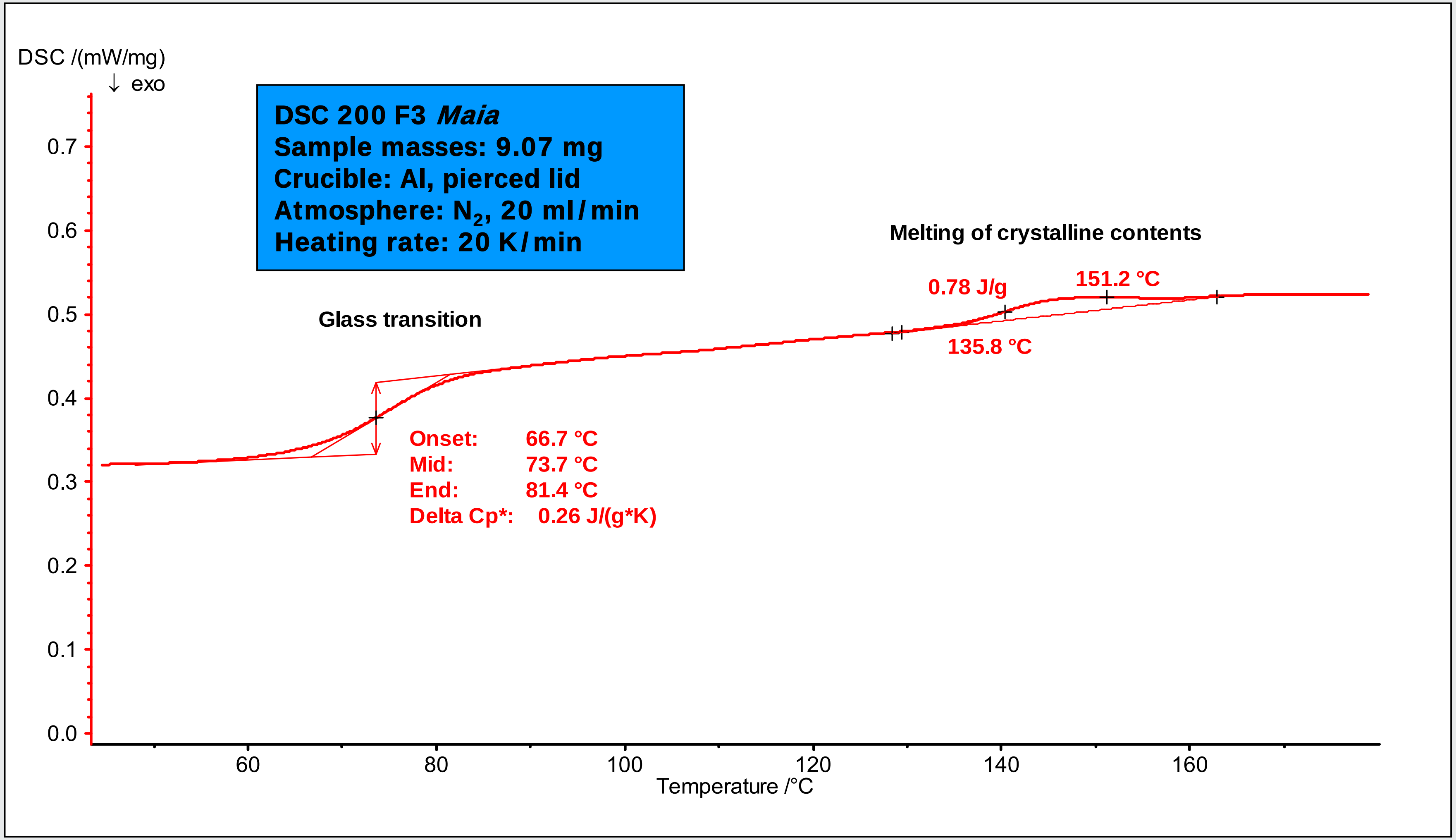


# PVC = Poly-Vinyl-Chloride



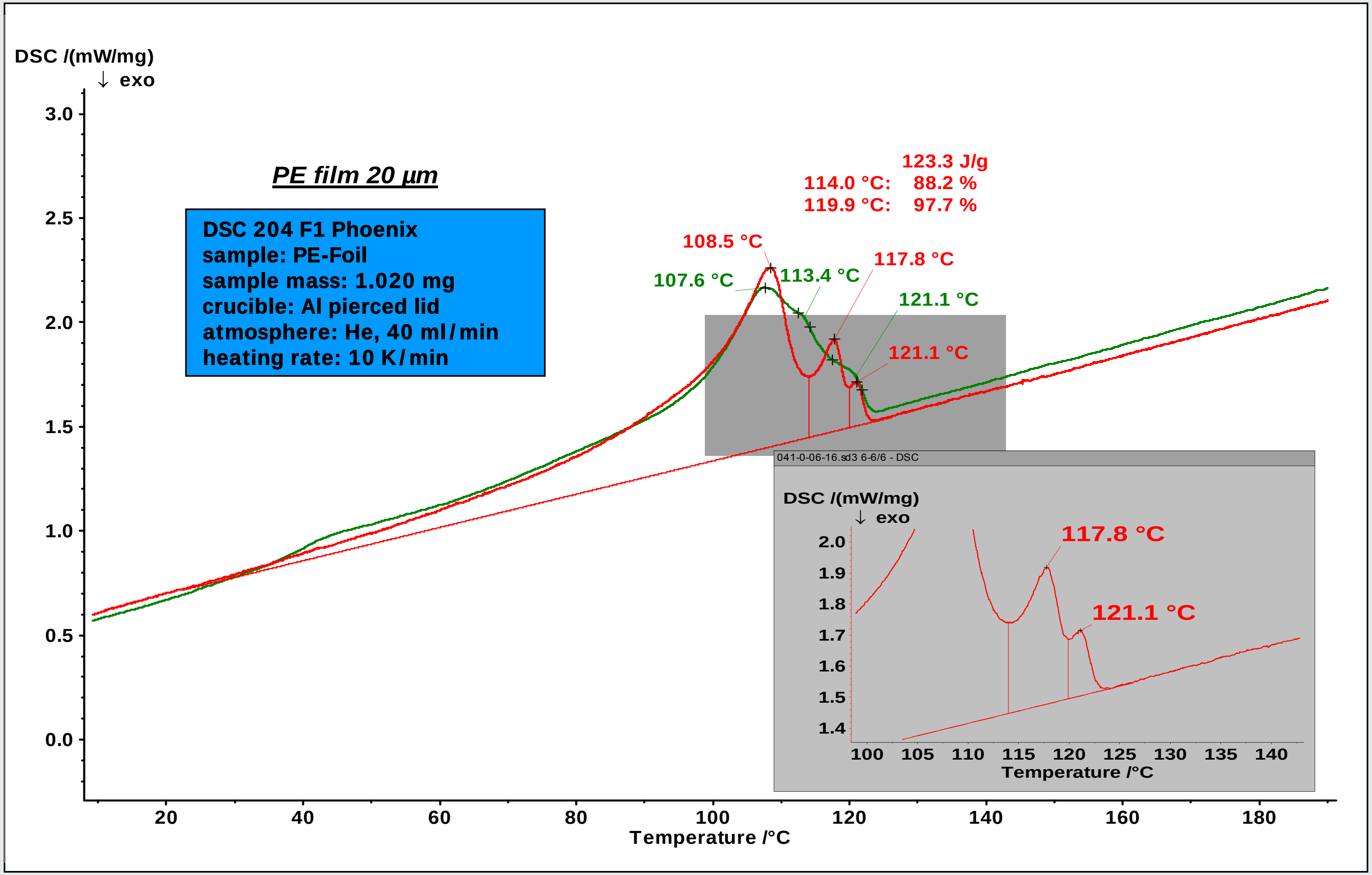


# Application – PVC

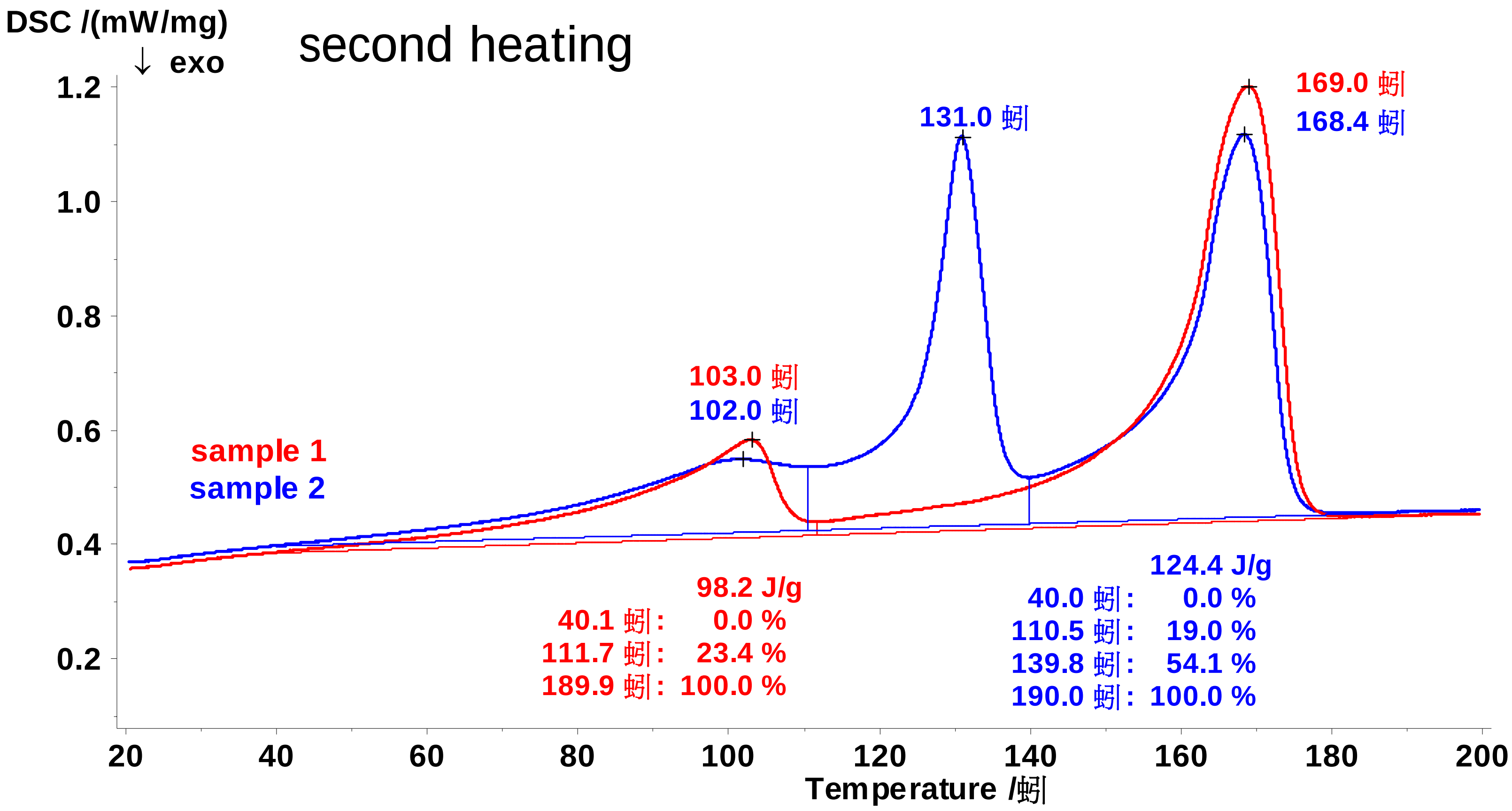


# Application $\tau$-Sensor: Separation of the Components of a PE-Blend

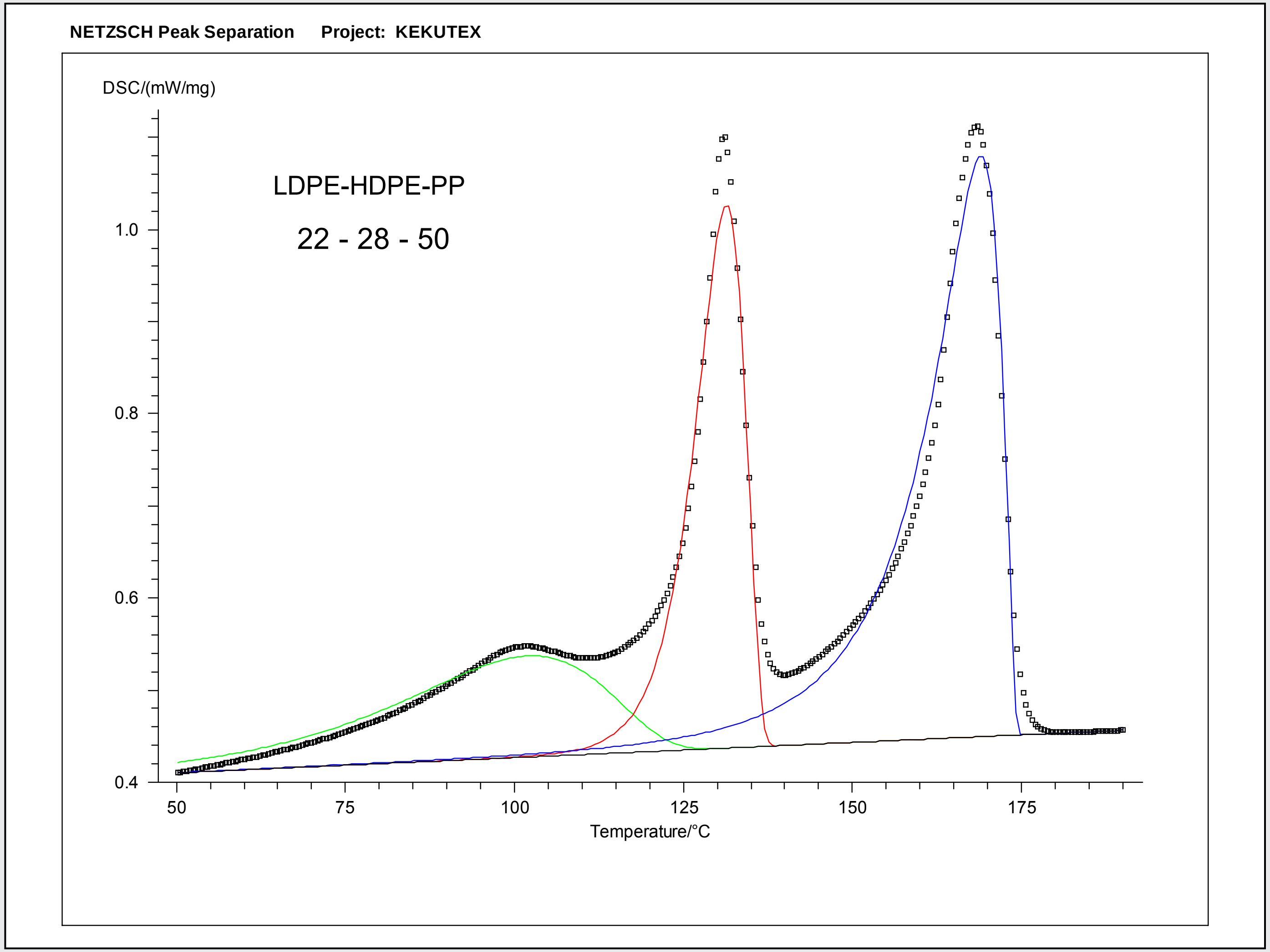
# Application τ-Sensor: Separation of the Components of a PE-Blend



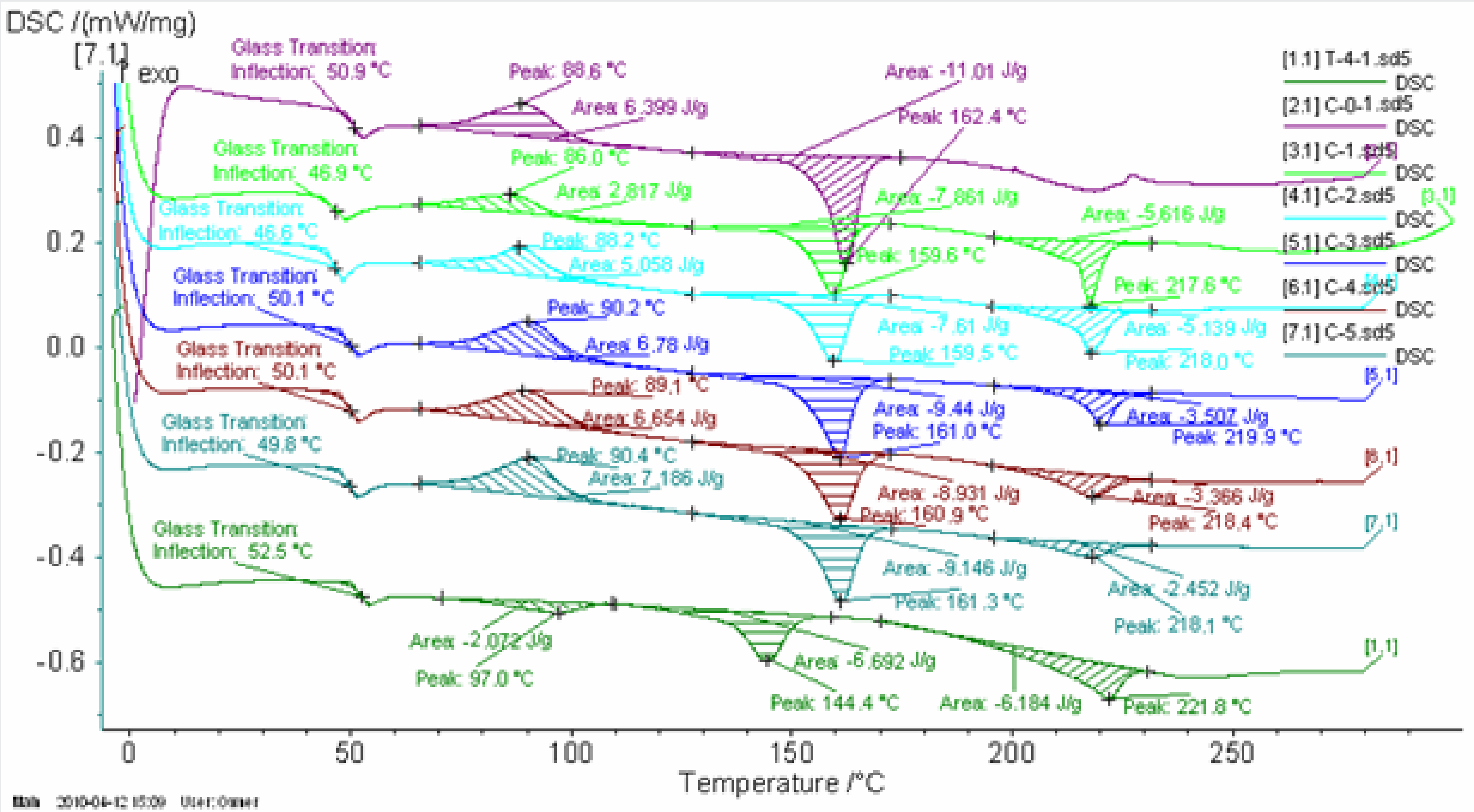
# LDPE-HDPE-PP


DSC /(mW/mg)
↓ exo
second heating
1.2
1.0
0.8
0.6
0.4
0.2
sample 1
sample 2
103.0 蚓
102.0 蚓
131.0 蚓
169.0 蚓
168.4 蚓
98.2 J/g
40.1 蚓: 0.0 %
111.7 蚓: 23.4 %
189.9 蚓: 100.0 %
124.4 J/g
40.0 蚓: 0.0 %
110.5 蚓: 19.0 %
139.8 蚓: 54.1 %
190.0 蚓: 100.0 %
20
40
60
80
100
120
140
160
180
200
Temperature /蚓

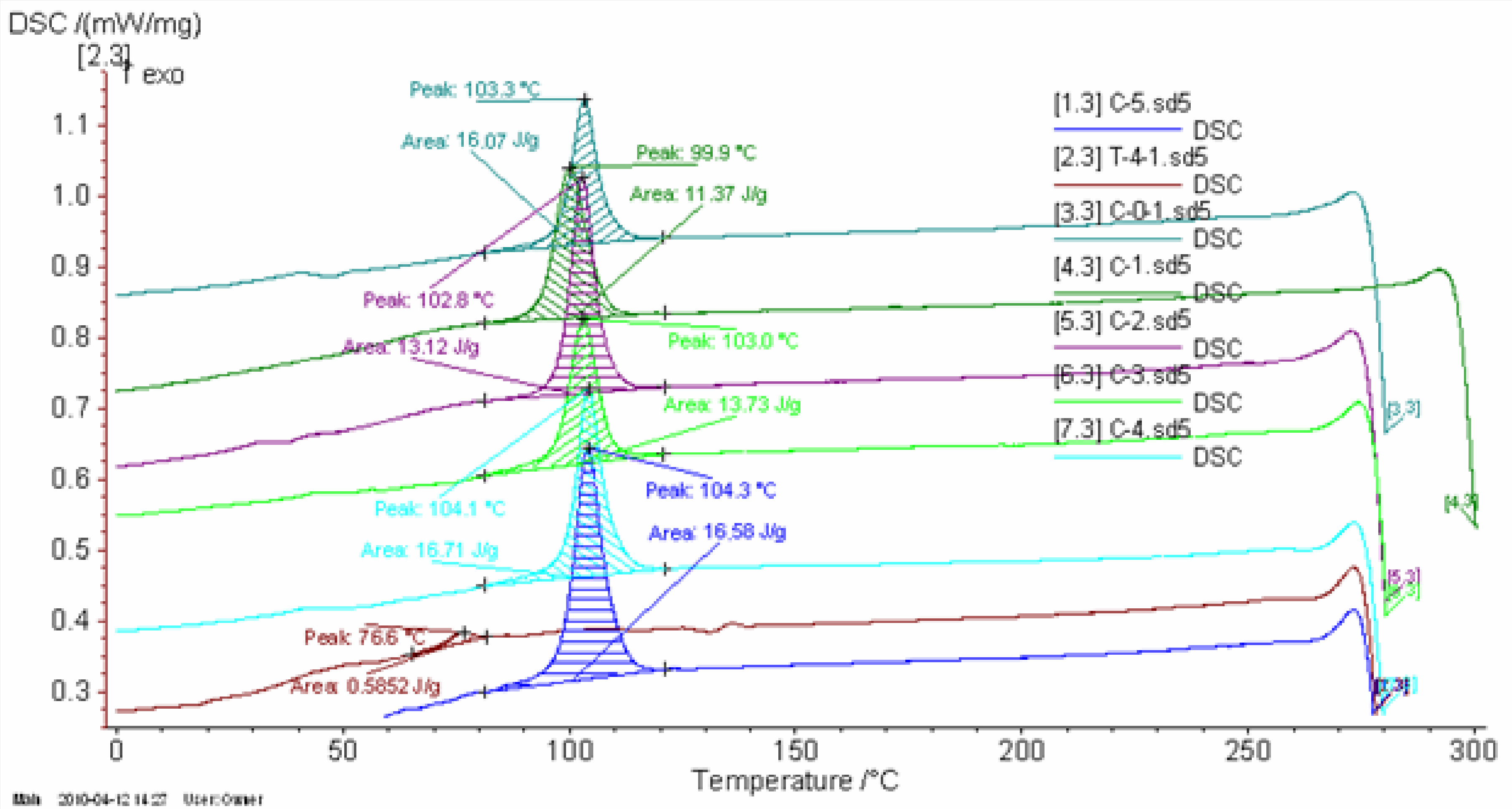
# LDPE-HDPE-PP (Peak Separation)



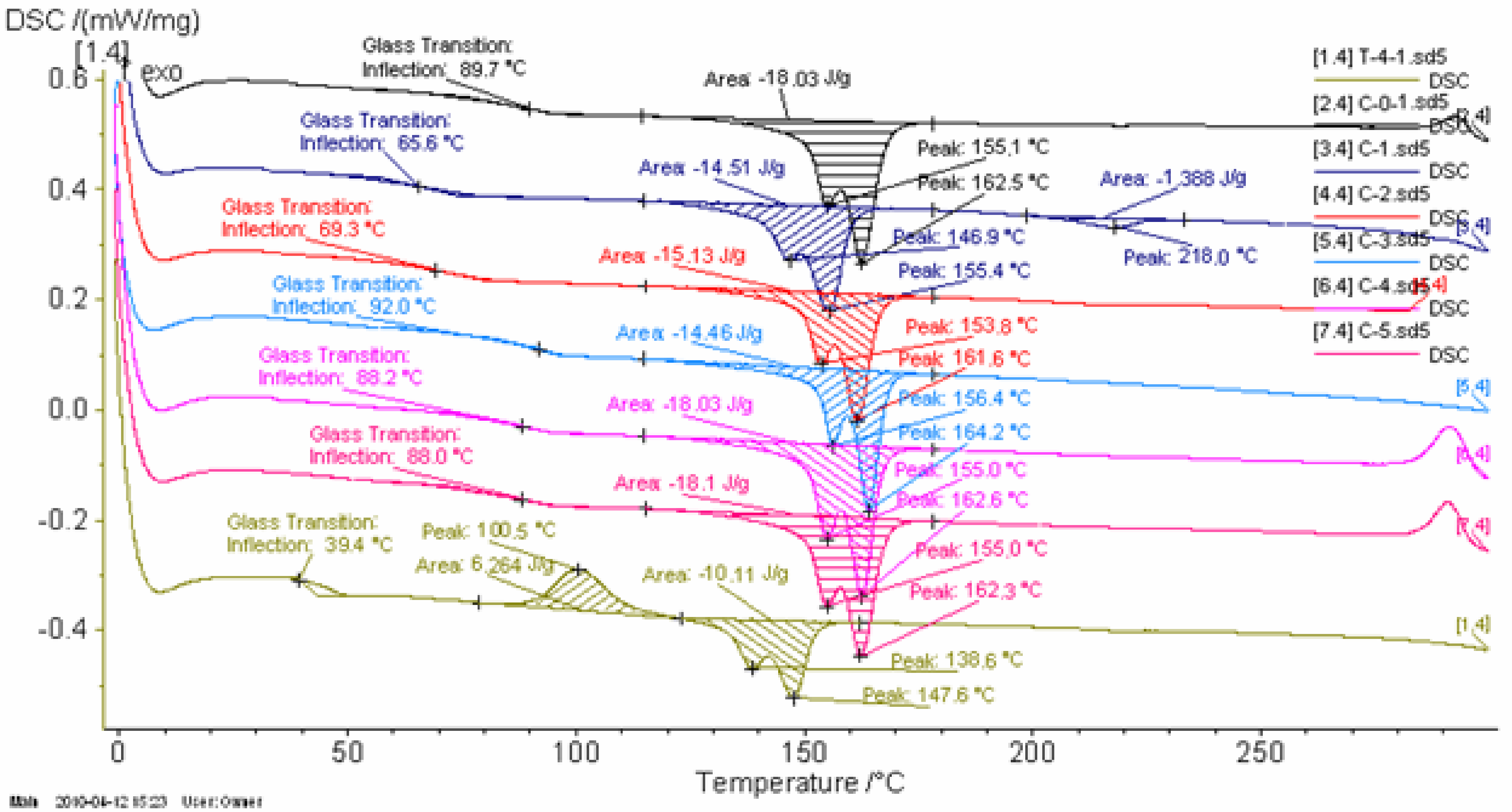
# DSC 不同配方及加工條件量測-Part 1
## (First Heating Rate 10°C/min )



# DSC 不同配方及加工條件量測-Part 2
## (First Cooling Rate 10°C/min )

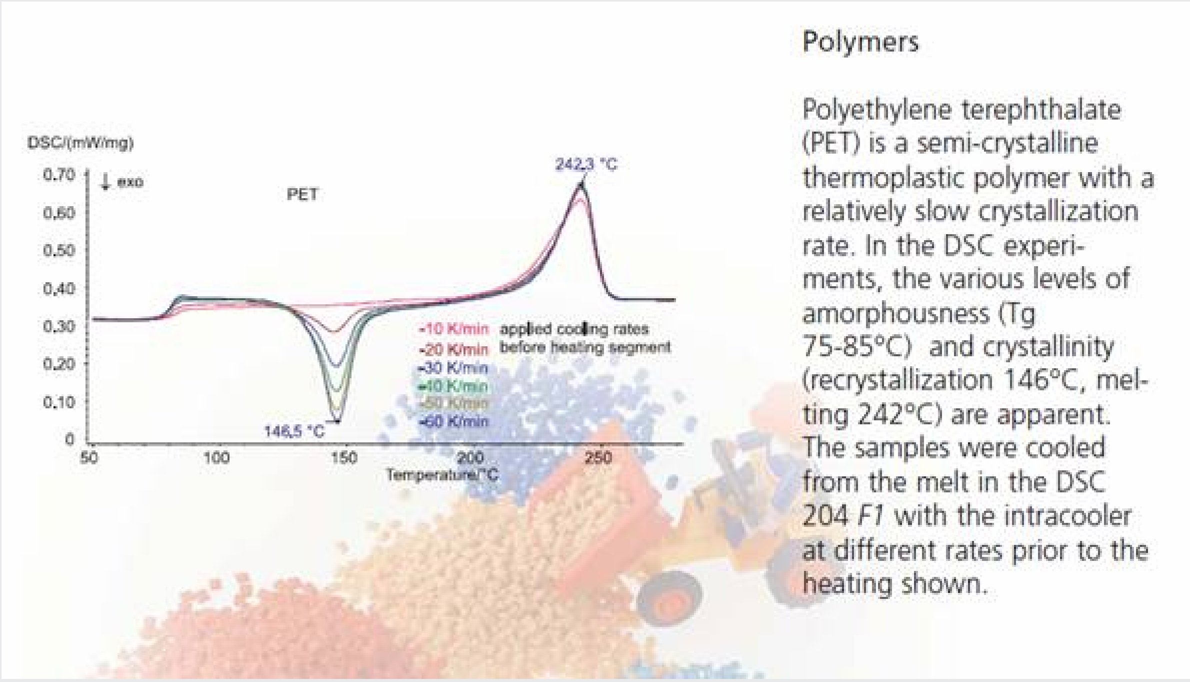


# DSC 不同配方及加工條件量測-Part 3
## (Second Heating Rate 10°C/min )

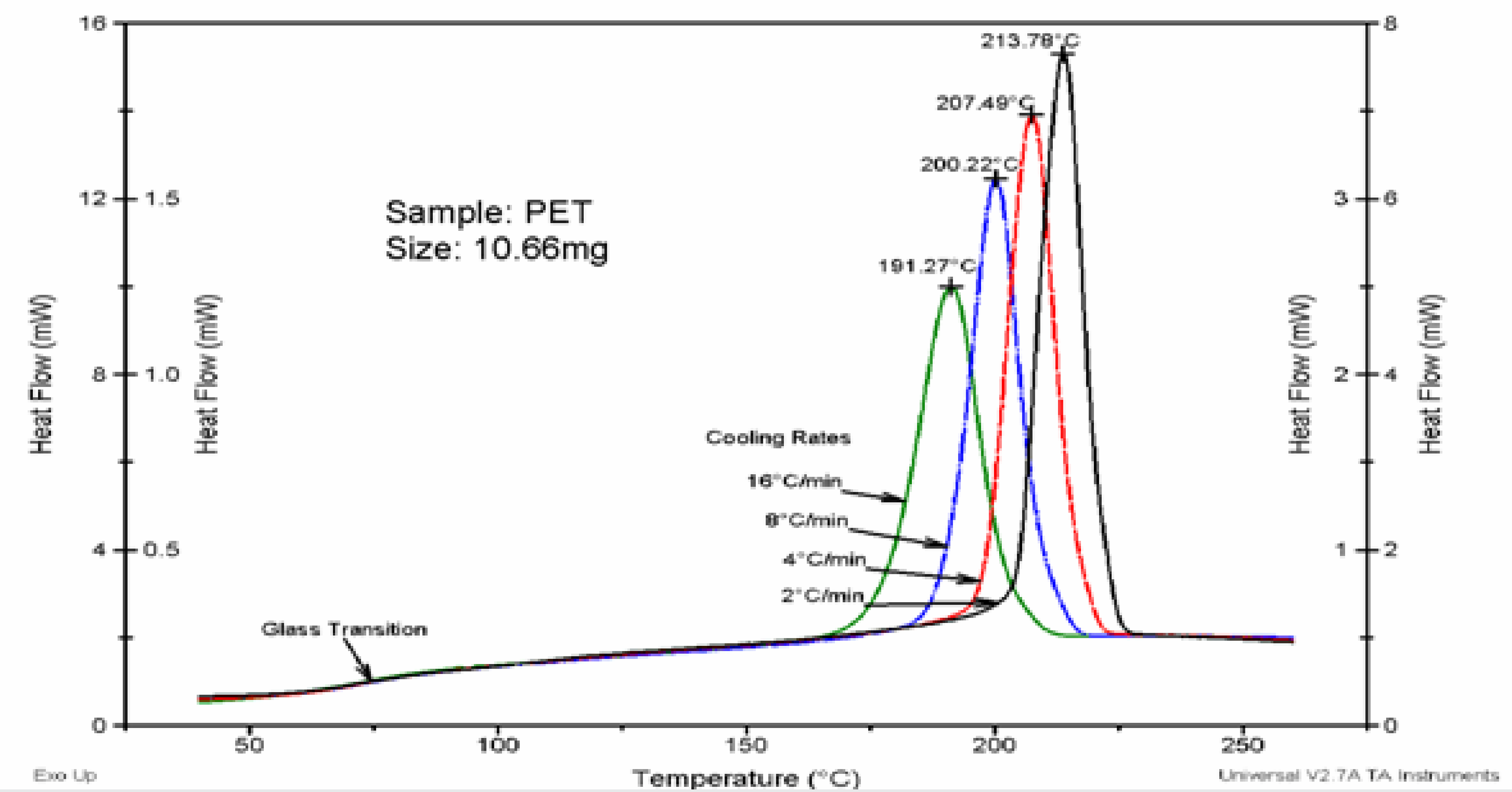


# DSC 不同配方及加工條件量測-Part 4 (結論說明 )

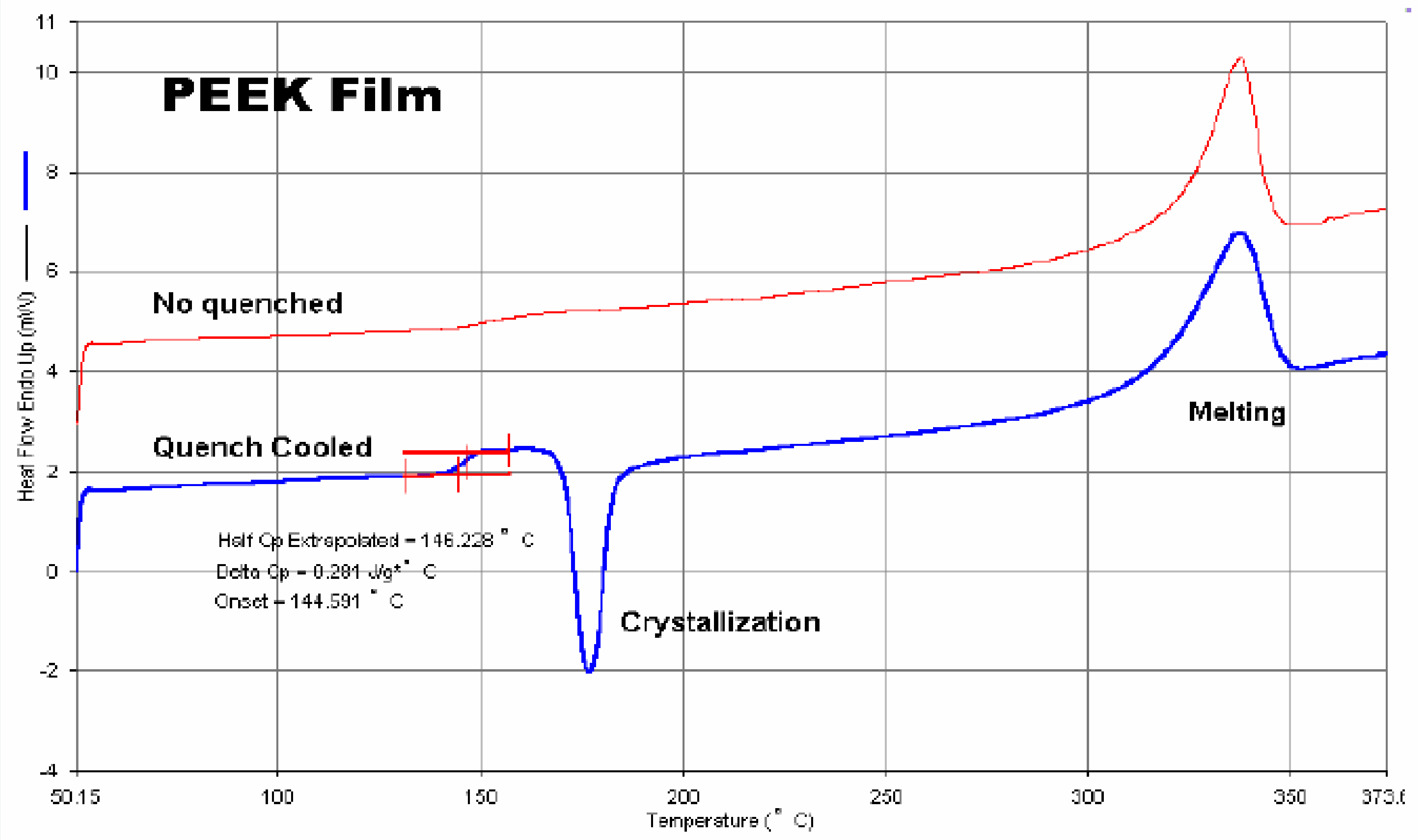
以下的圖是PET以不同的冷卻速率做實驗再做第二次加熱的曲線,
紅色曲線(10°C/min cooling rate)爲例,冷卻過程樣品會有結晶現象,再次加熱時就看不到再結晶現象,
靛藍色曲線(60°C/min cooling rate)爲例,冷卻過程樣品由於冷卻速率太快沒有結晶現象,再次加熱時就看到再結晶現象,
其它的不同冷卻速率再次加熱後所得到不同的再次結晶熱.
至於貴司的樣品T-4在冷卻過程看不到(很小)冷卻結晶,可能10°C/min對此T-4的樣品冷卻速率太快,來不及結晶,
所以在第二次加熱時會看到再結晶現象.



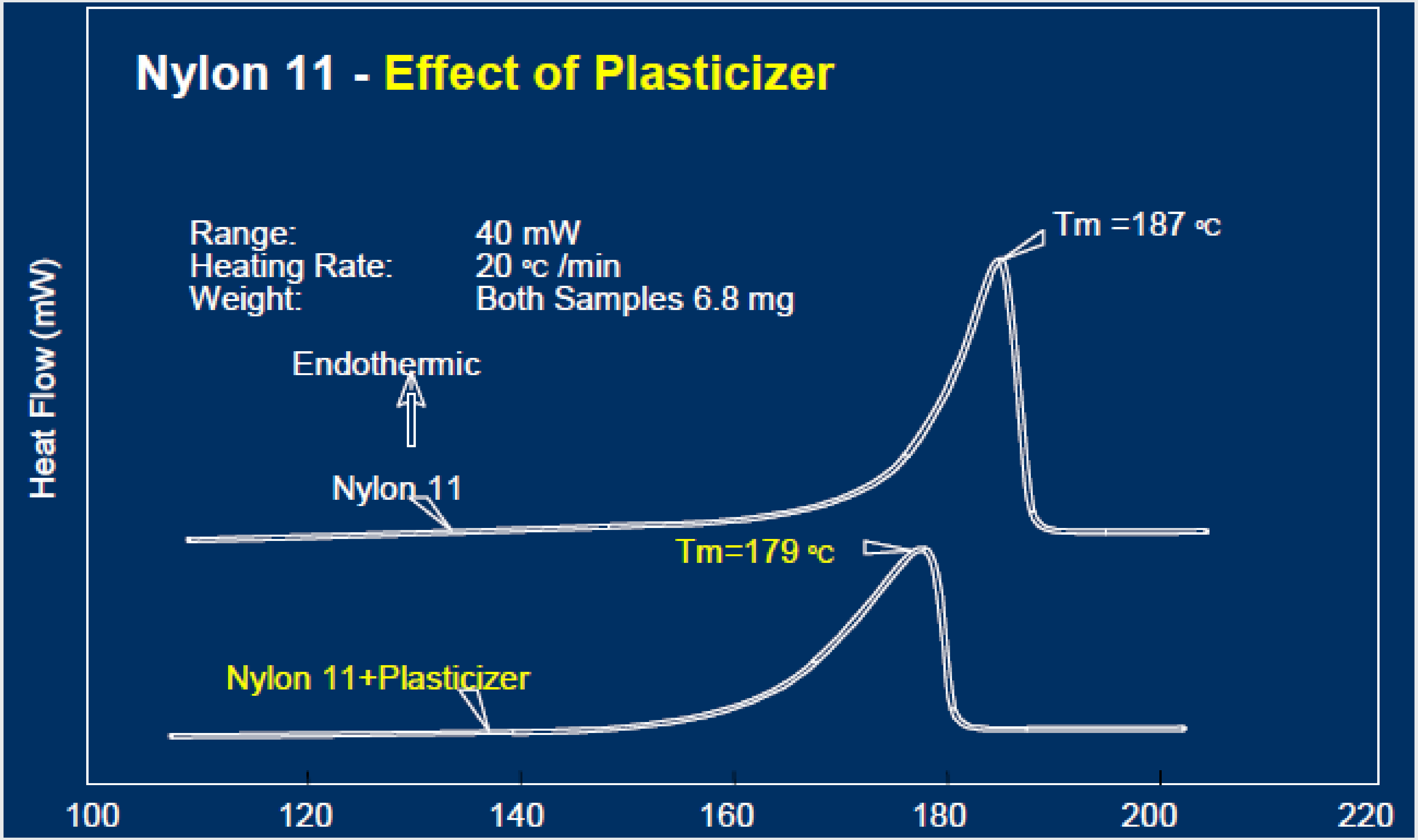
# 冷却速率对 PET 结晶的影响



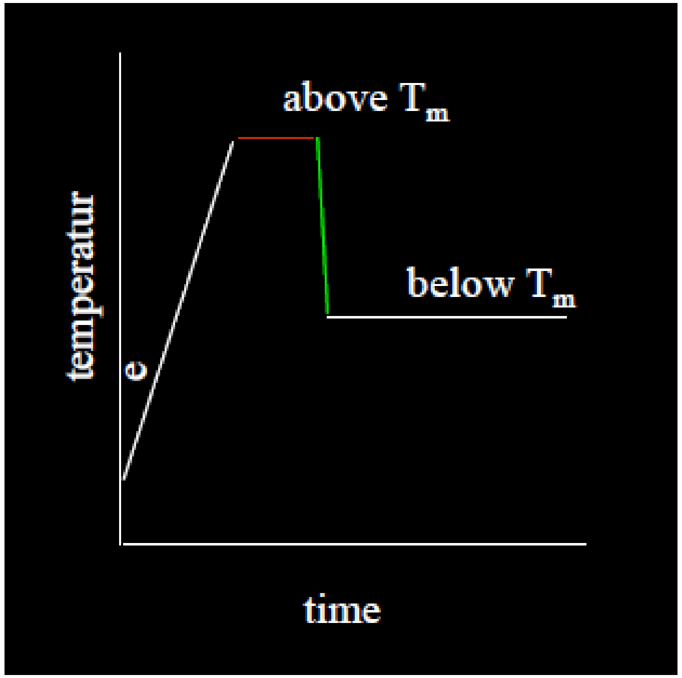
# *Tg of Polymers–history of heat process can alter Tg*



## *Analysis of Tm–effect of plasticizer*

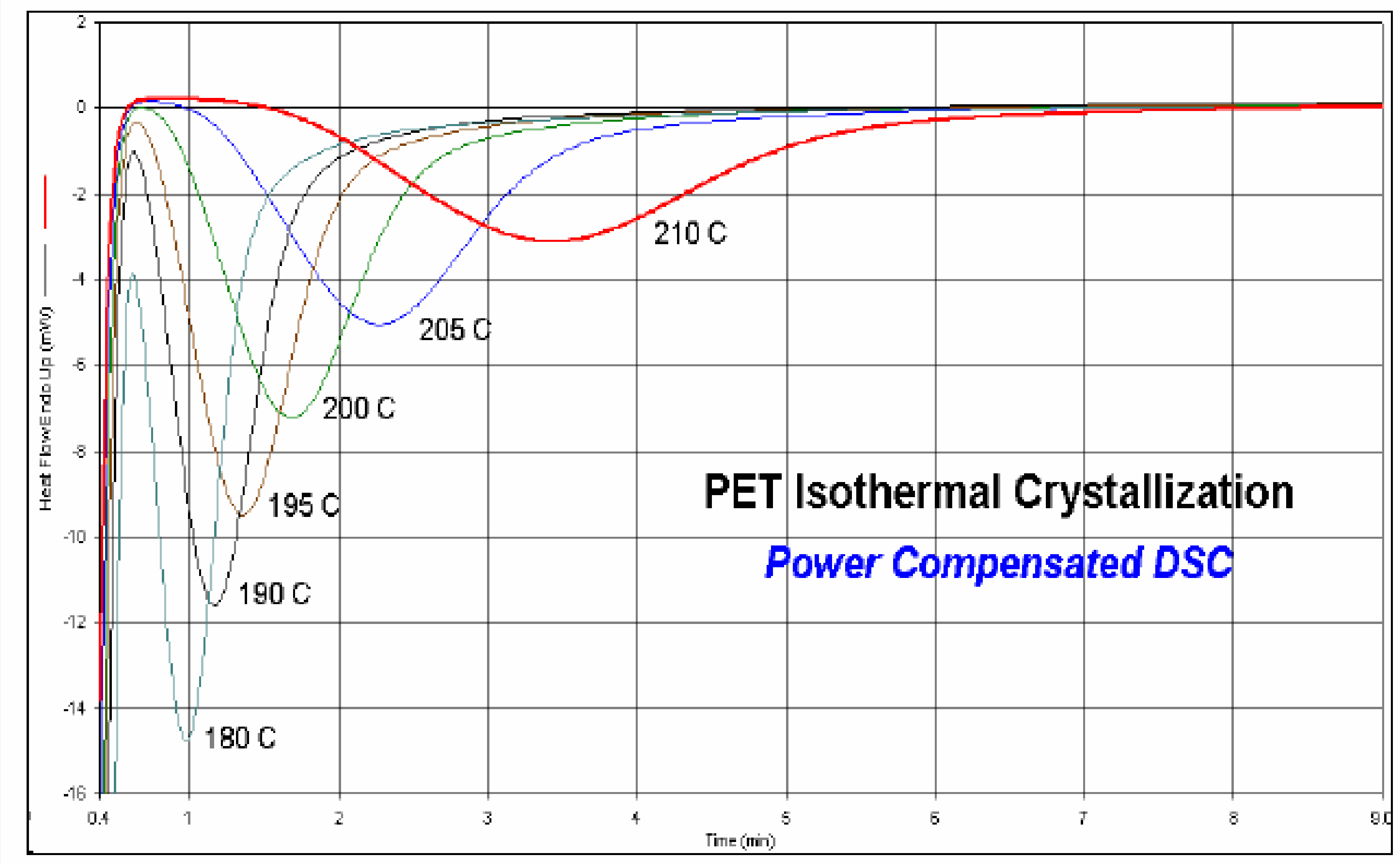


# *Isothermal test for crystallinity*

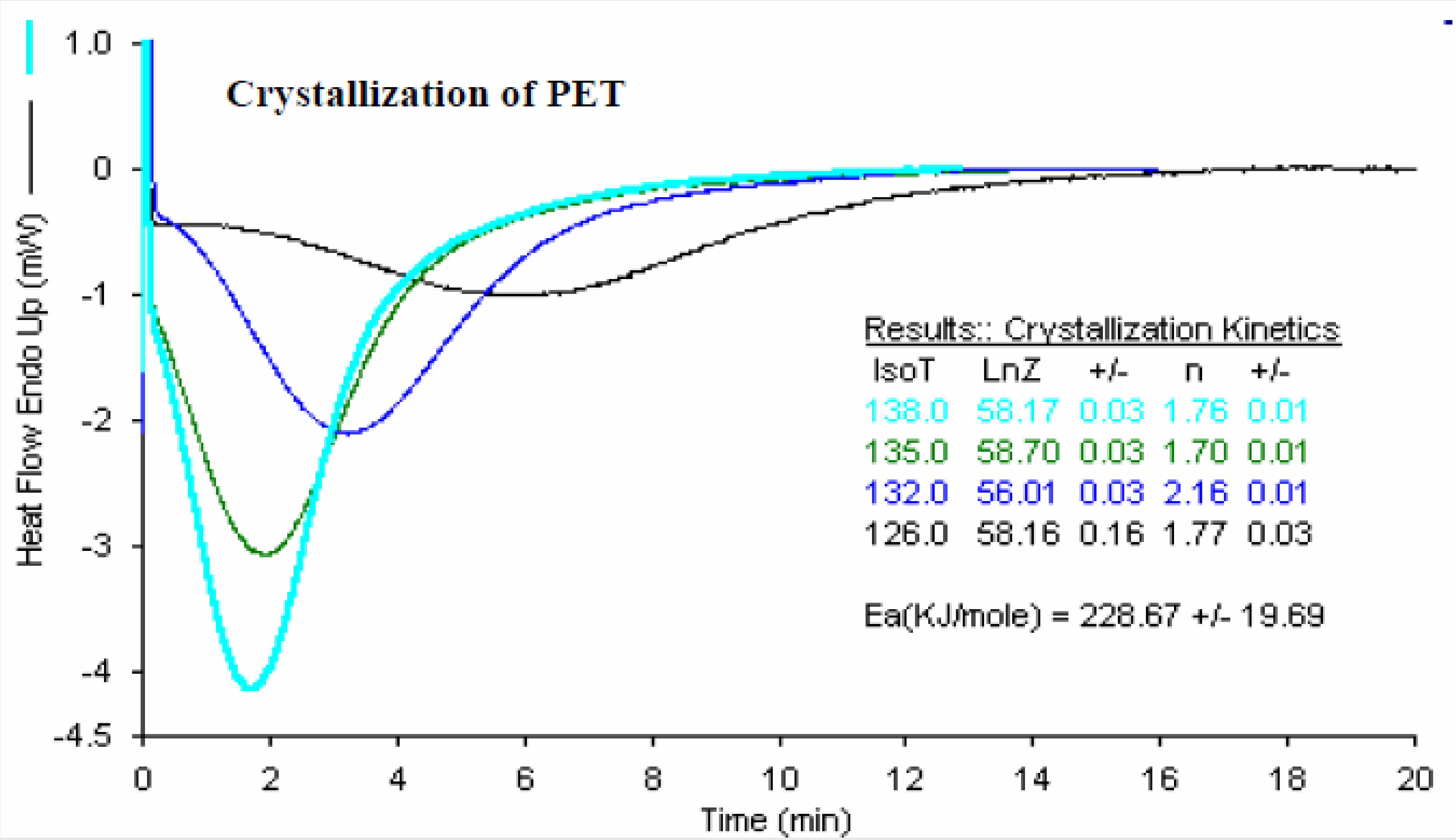



- *Heat the sample over Tm for a period for complete melting*
- *Quench the sample at T< Tm*
- *Measure heat change in sample*

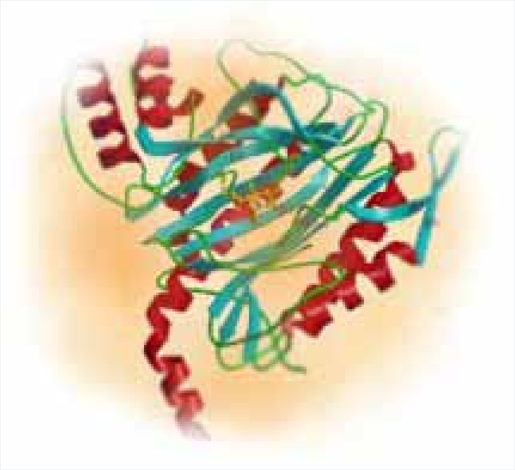
# *Isothermal test for crystallinity*



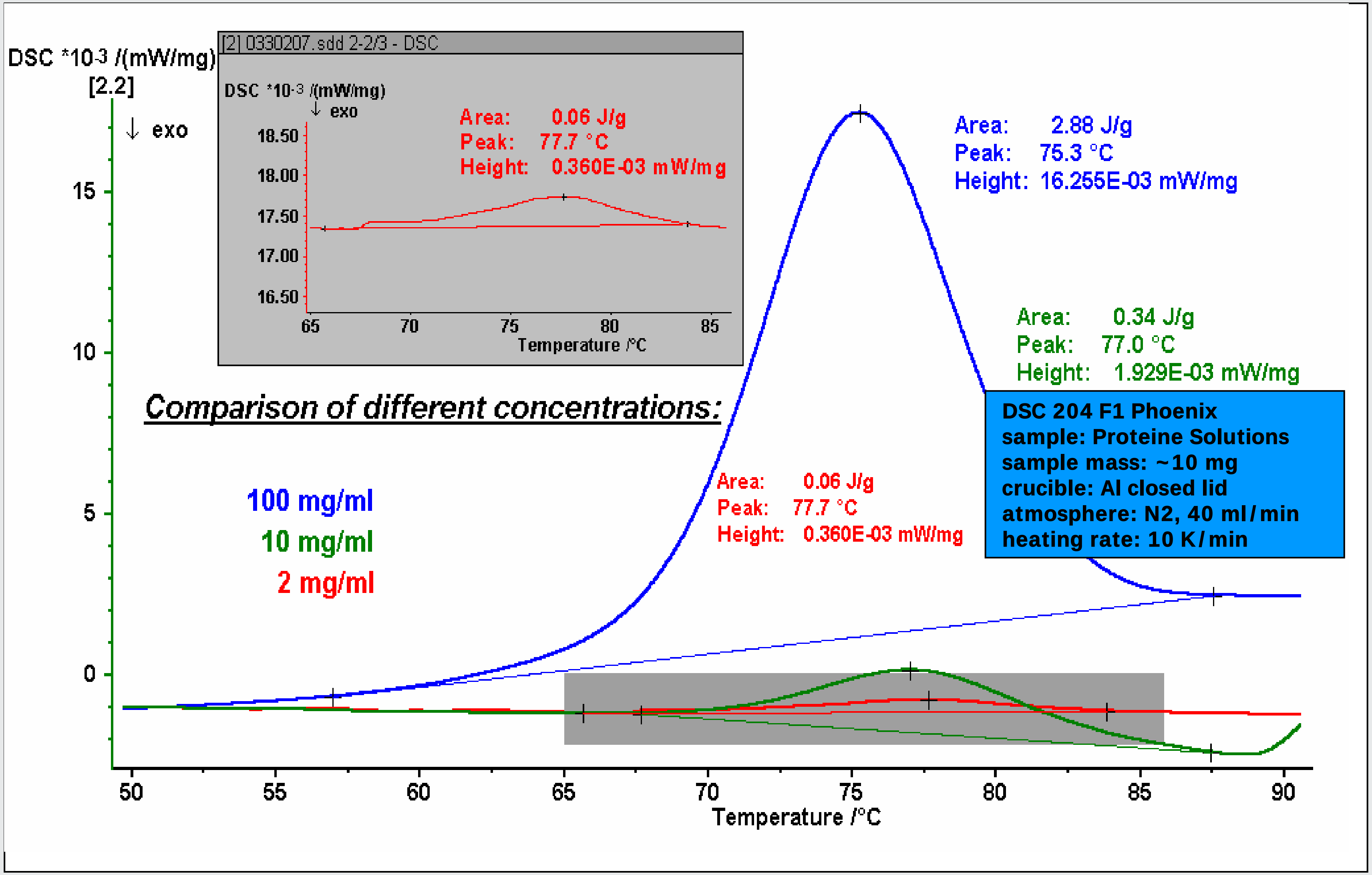
# 動力學分析-- Isothermal Kinetics(Option)



# Application µ-Sensor: Denaturation of Proteine Solutions



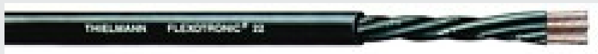

# Application µ-Sensor: Denaturation of Proteine Solutions



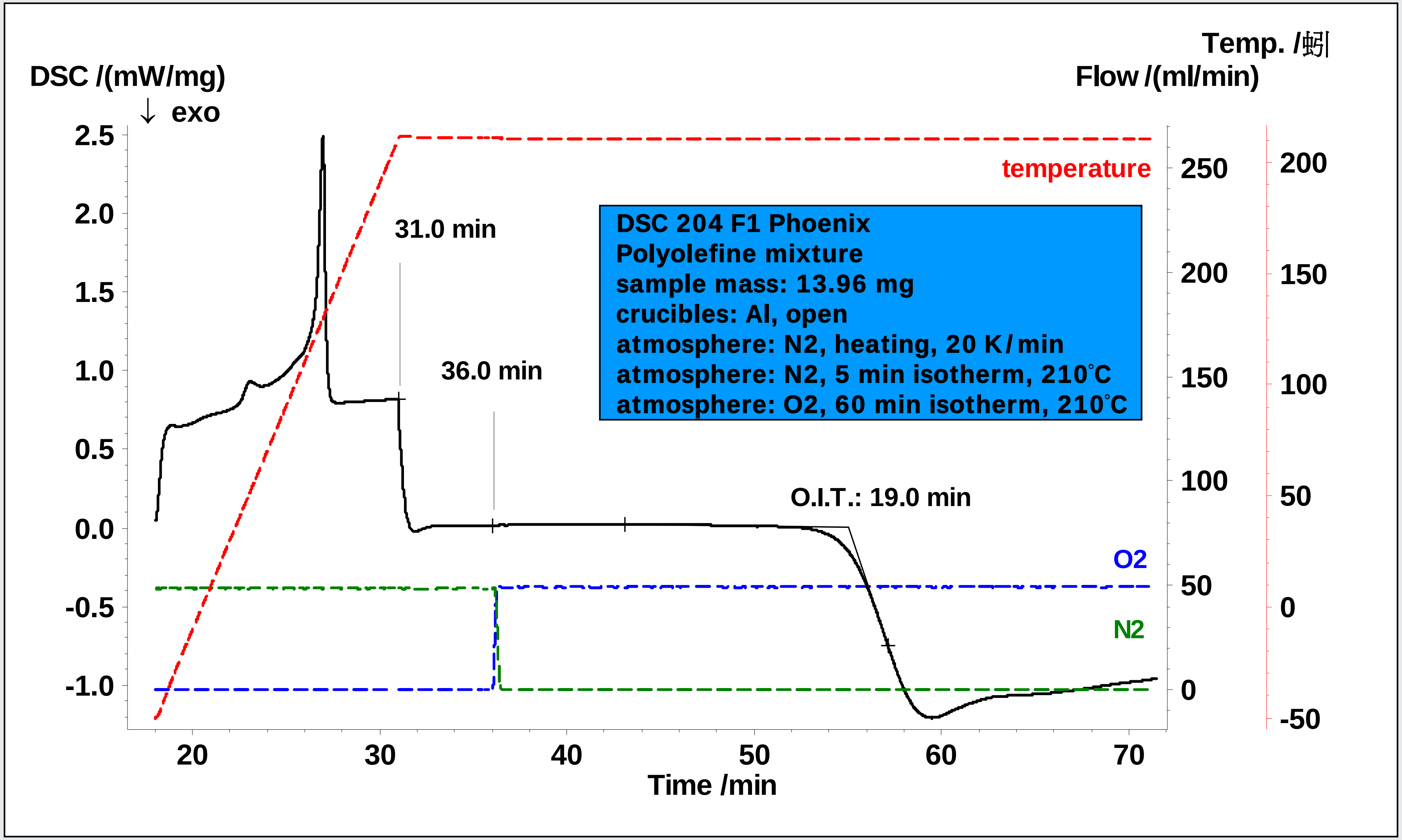
# Application – HDPE (O.I.T.-Tests)

**The DSC 200 F3 *Maia*® allows determination of the oxidative induction time (O.I.T.) according to well-established standards (ASTM D 38 95, ASTM D 6186, EN 728 und ISO 11357-3).**

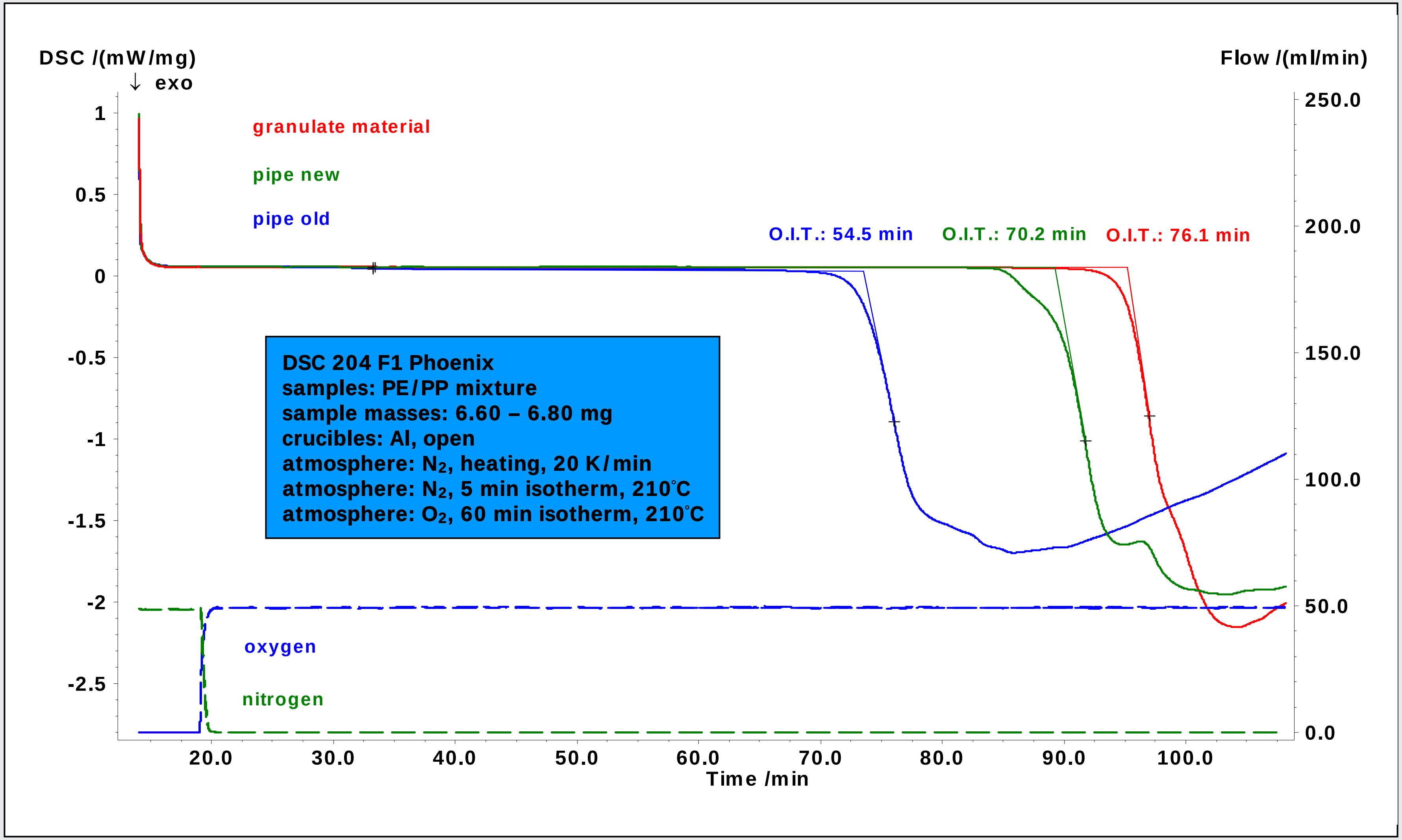


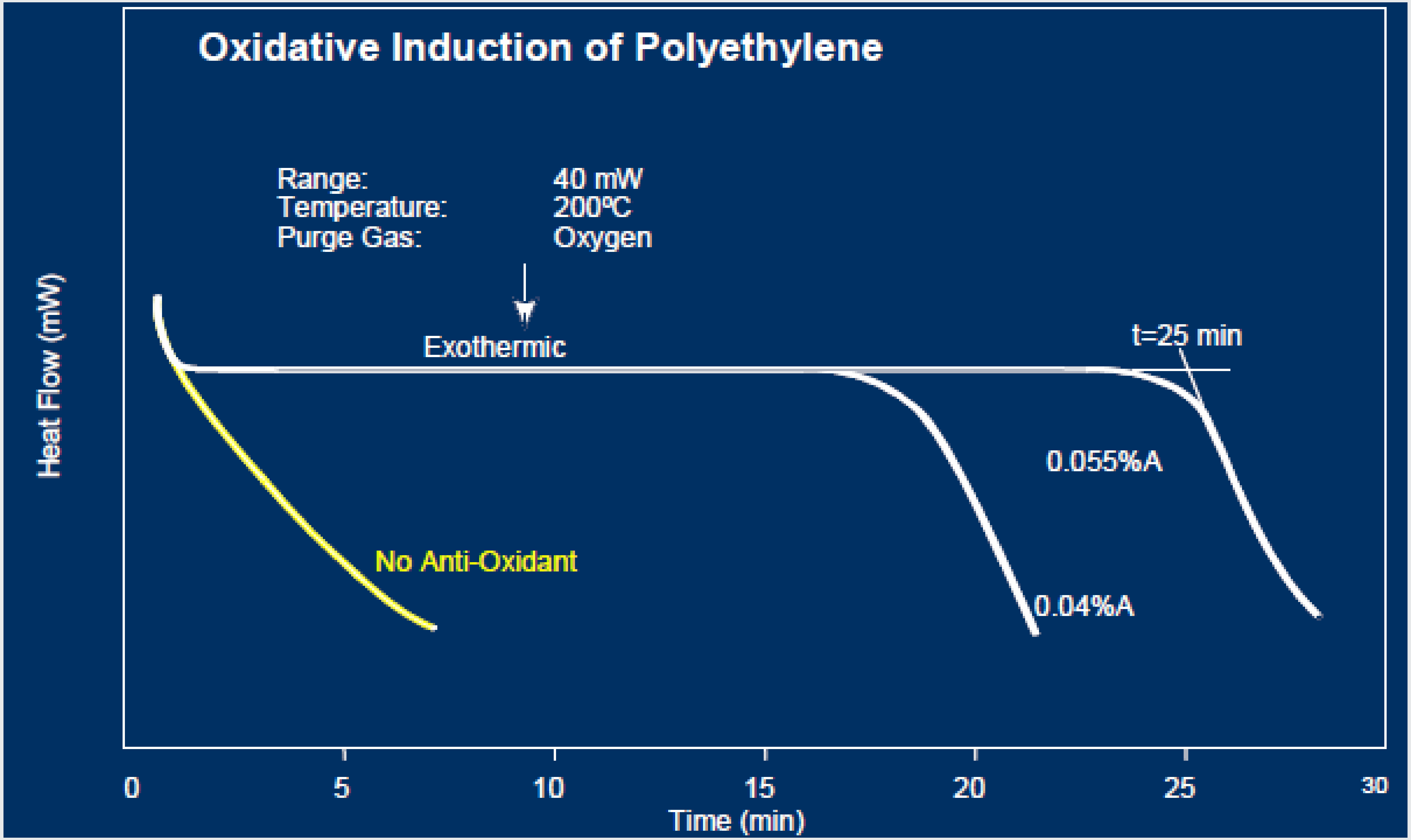
# Oxidation Induction Time (O.I.T.)



# Oxidation Induction Time (O.I.T.) Application on a PE/PP-Pipe Material

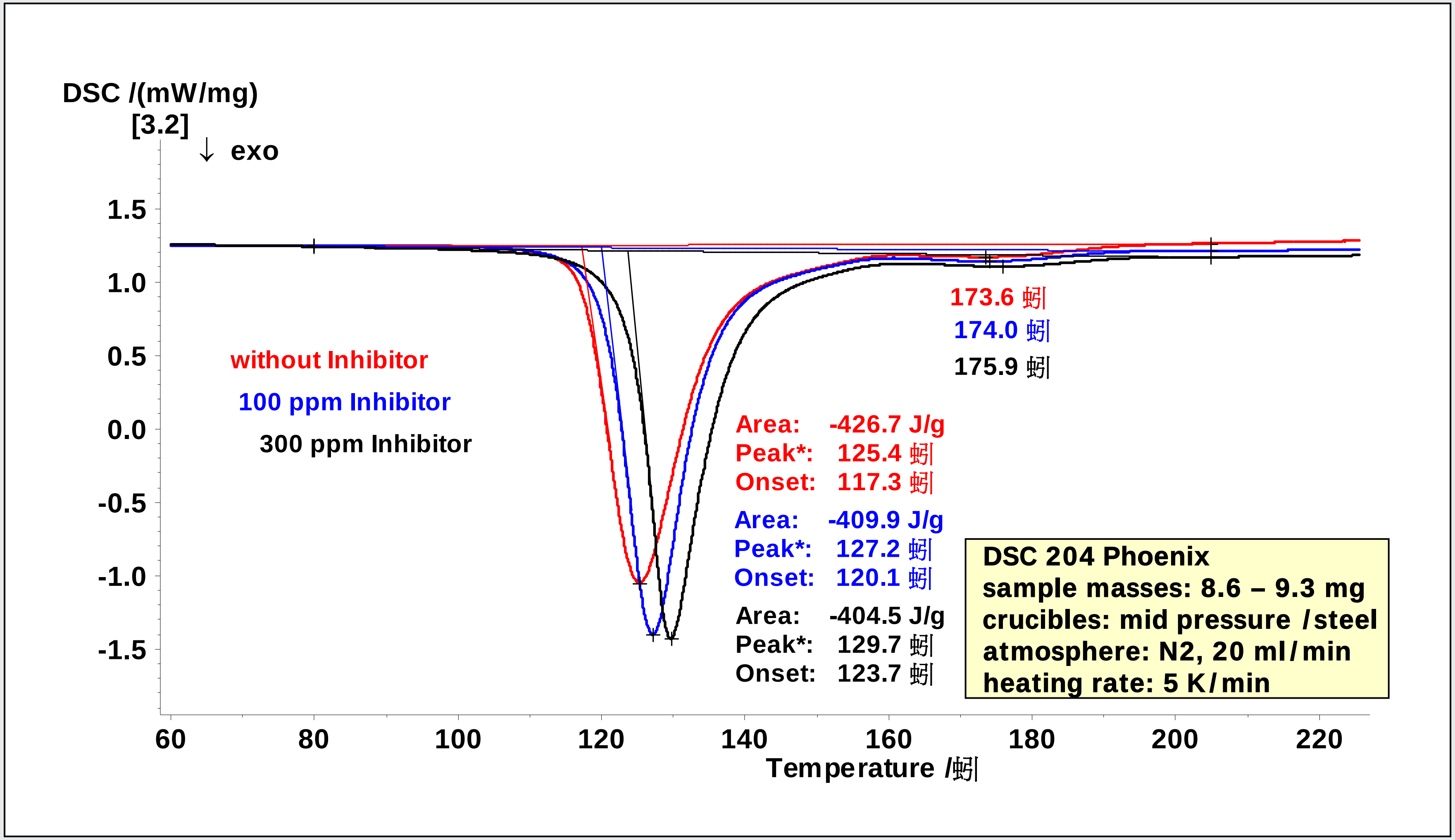


# *Oxidative induction of PE (To know the anti-oxidation capability)*

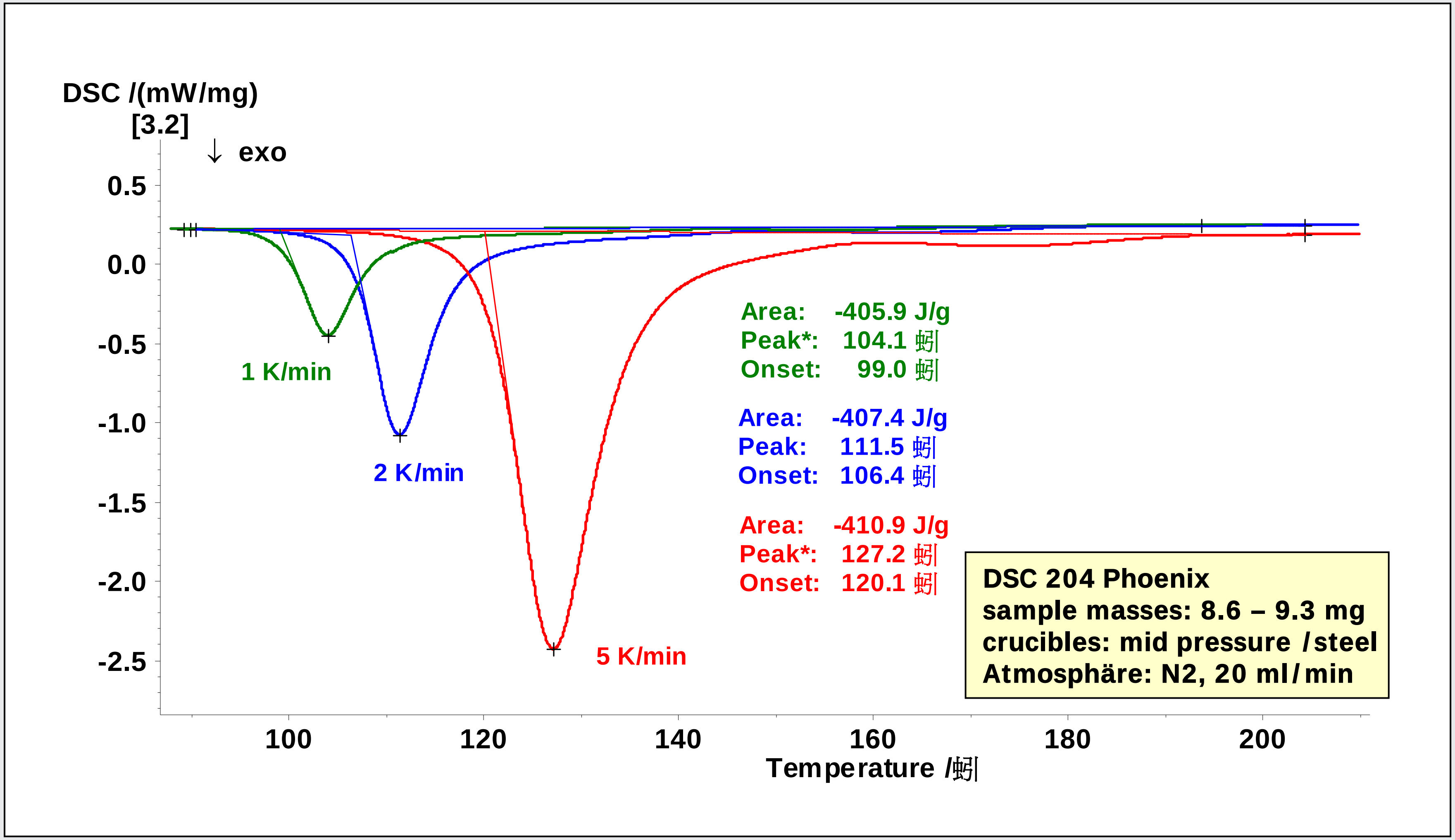


# Wind power plants

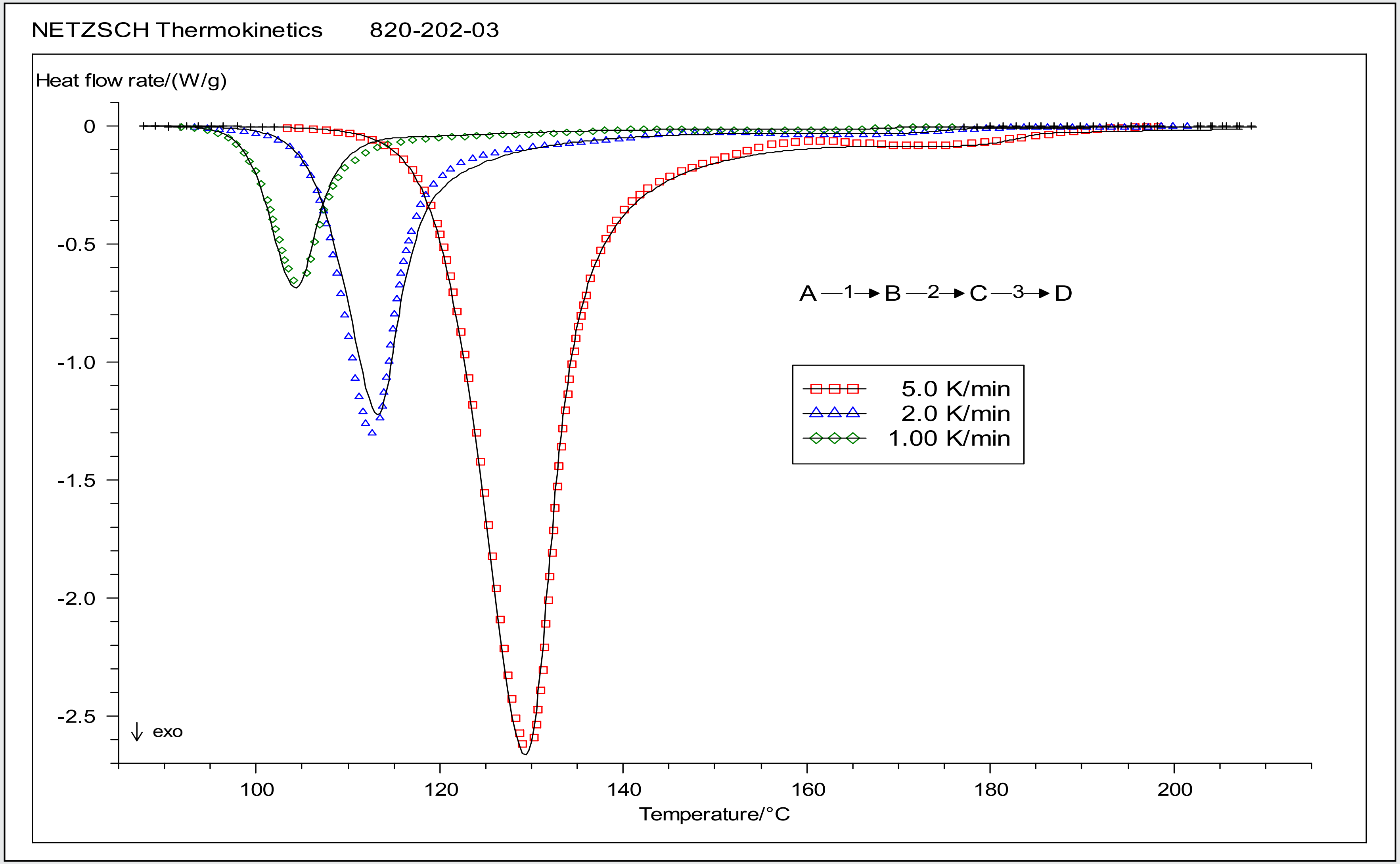
# Curing of Polyester Resin



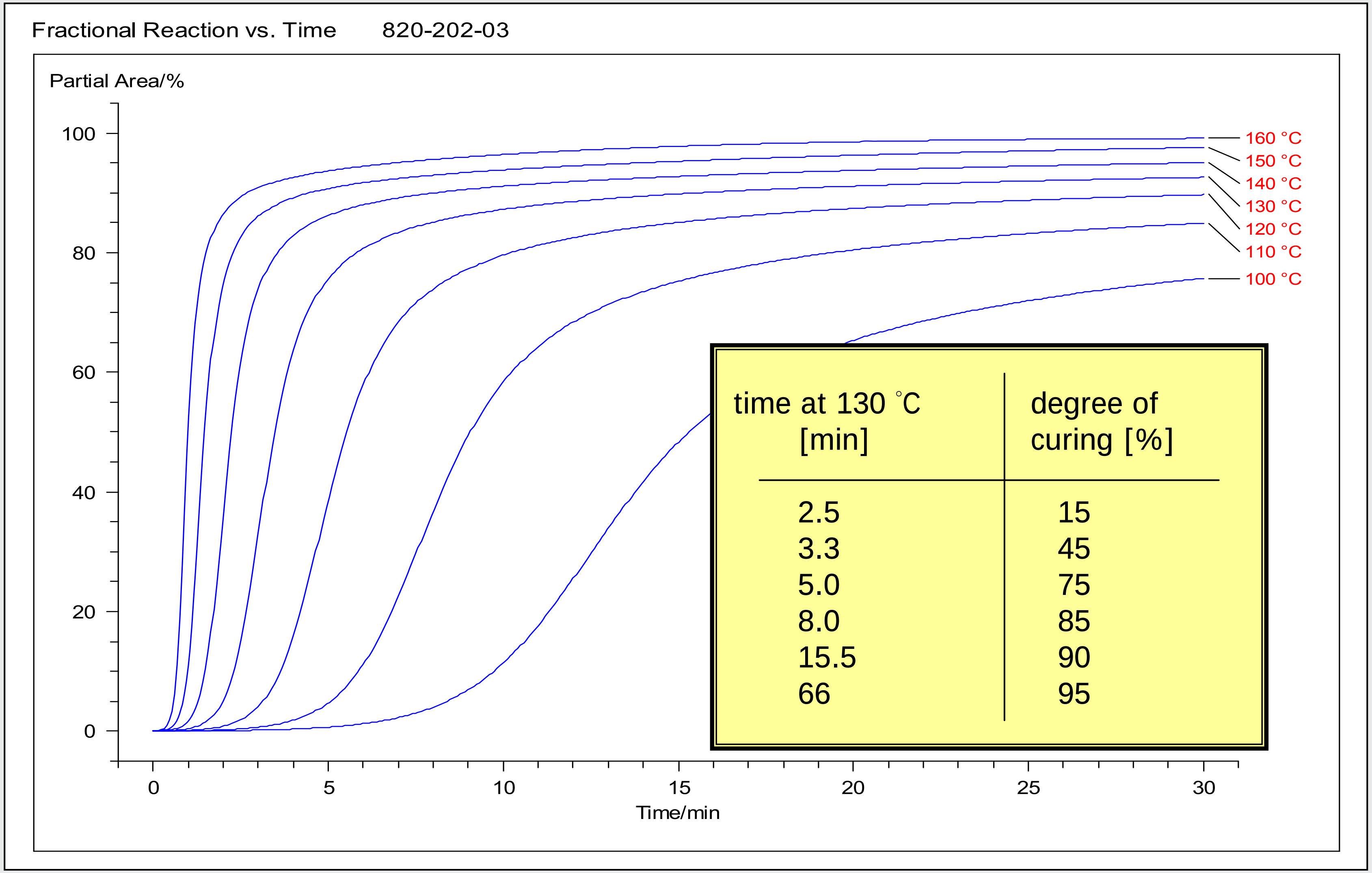
# Curing of Polyester Resin



# Curing of Polyester Resin (Thermokinetics)



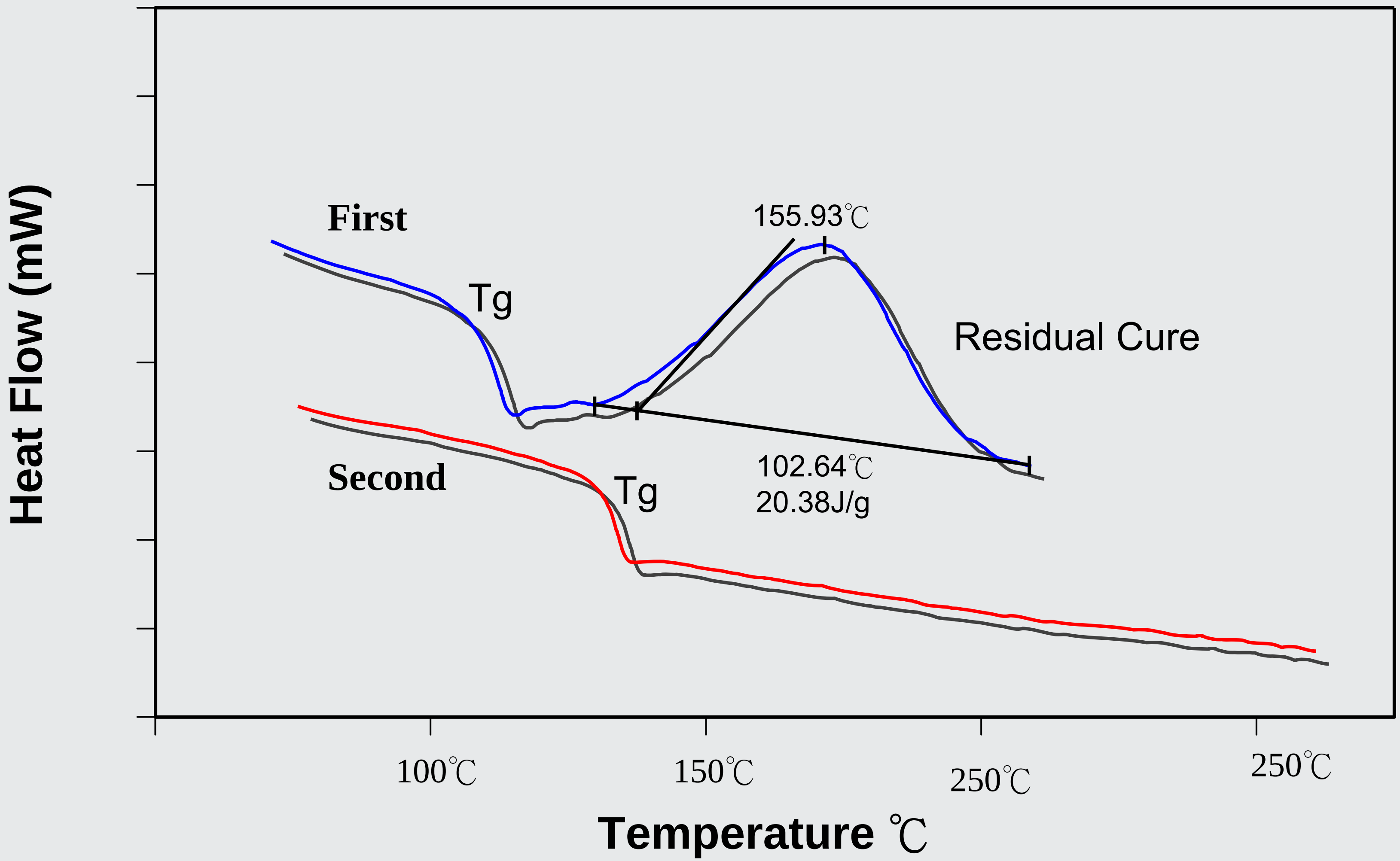
# Curing of Polyester Resin (Predictions)



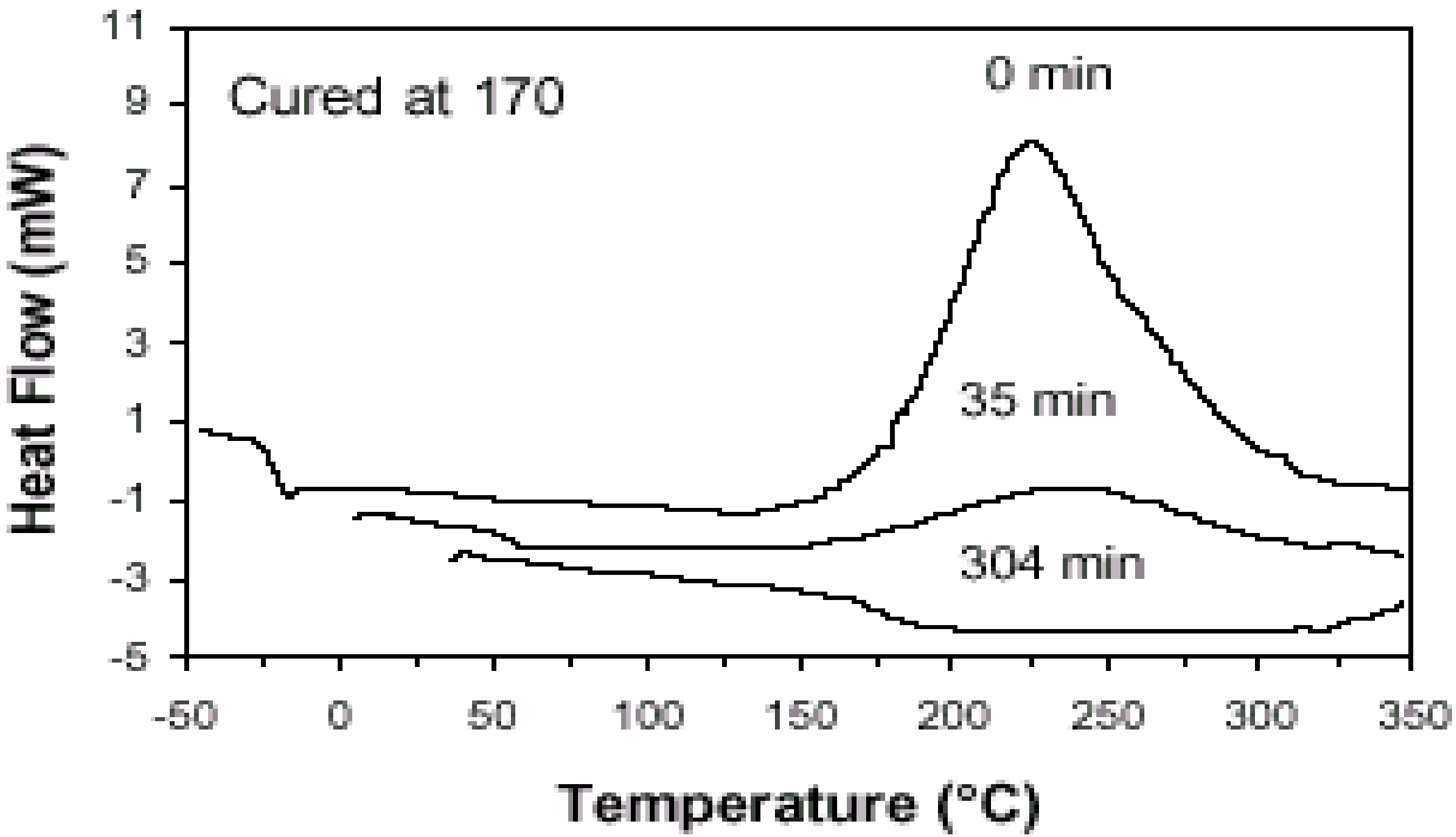

| time at 130 °C [min] | degree of curing [%] |
|---|---|
| 2.5 | 15 |
| 3.3 | 45 |
| 5.0 | 75 |
| 8.0 | 85 |
| 15.5 | 90 |
| 66 | 95 |

# Thermal Analysis on Curing Monitoring of Thermosetting Resins

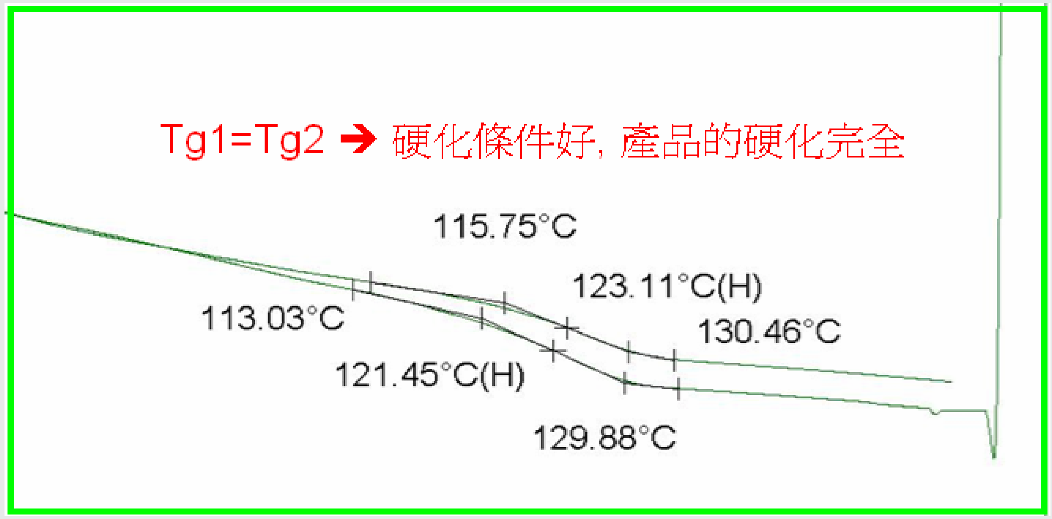
# Thermoset: Comparison of First and Second Heating Runs



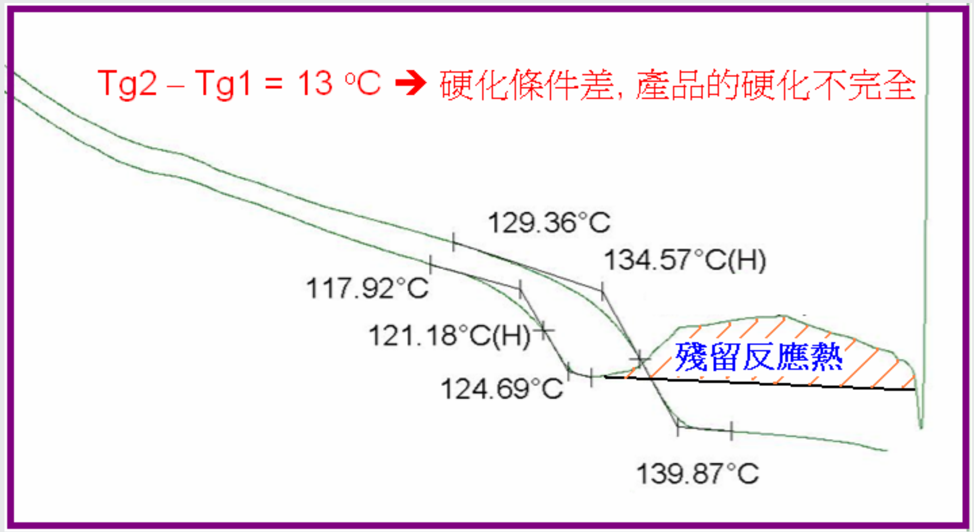
# Thermoset:Isothermal Temp 170 Deg C
## Different IsOthermal test results comparison



# Tg1/Tg2 試驗法

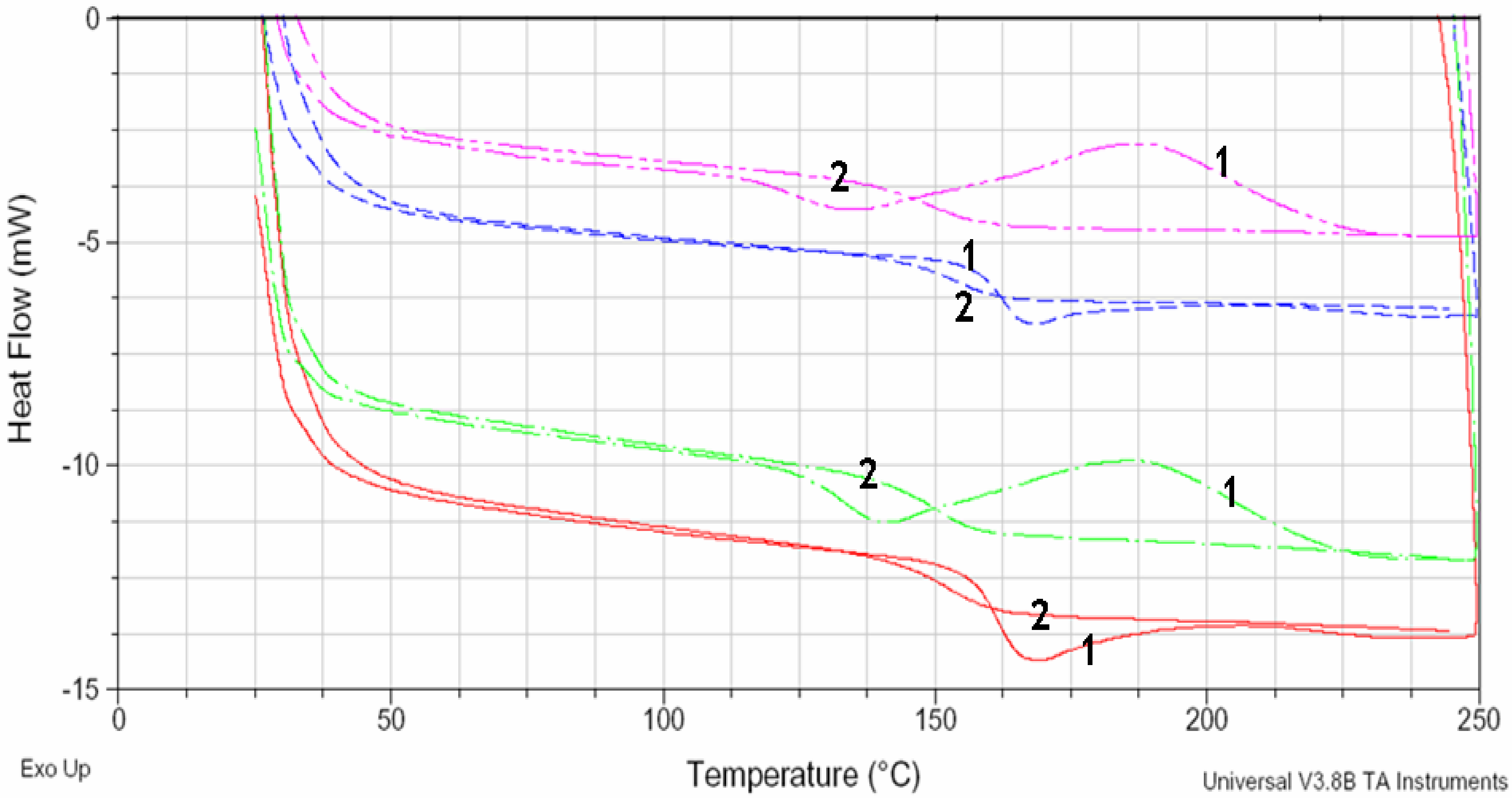


Tg1=Tg2 ➔ 硬化條件好, 產品的硬化完全
115.75°C
123.11°C(H)
113.03°C
130.46°C
121.45°C(H)
129.88°C

Tg2 – Tg1 = 13 °C ➔ 硬化條件差, 產品的硬化不完全
129.36°C
134.57°C(H)
117.92°C
121.18°C(H)
殘留反應熱
124.69°C
139.87°C

# 利用Tg1/Tg2的方式評價Epoxy是否完全固化?



# 常用DSC決定熱固型(Thermoset)材料是否固化完全(Full Curing)的方法：Tg1/Tg2 試驗法

Step1 - 以20OC/min 掃瞄求出 Tg1

Step2 - 在一足夠高的溫度下 (如175 $^{O}$C) 下烘烤 15min 以保證完全反應

Step3 - 再次以 20$^{O}$C/min 掃瞄求出 Tg2

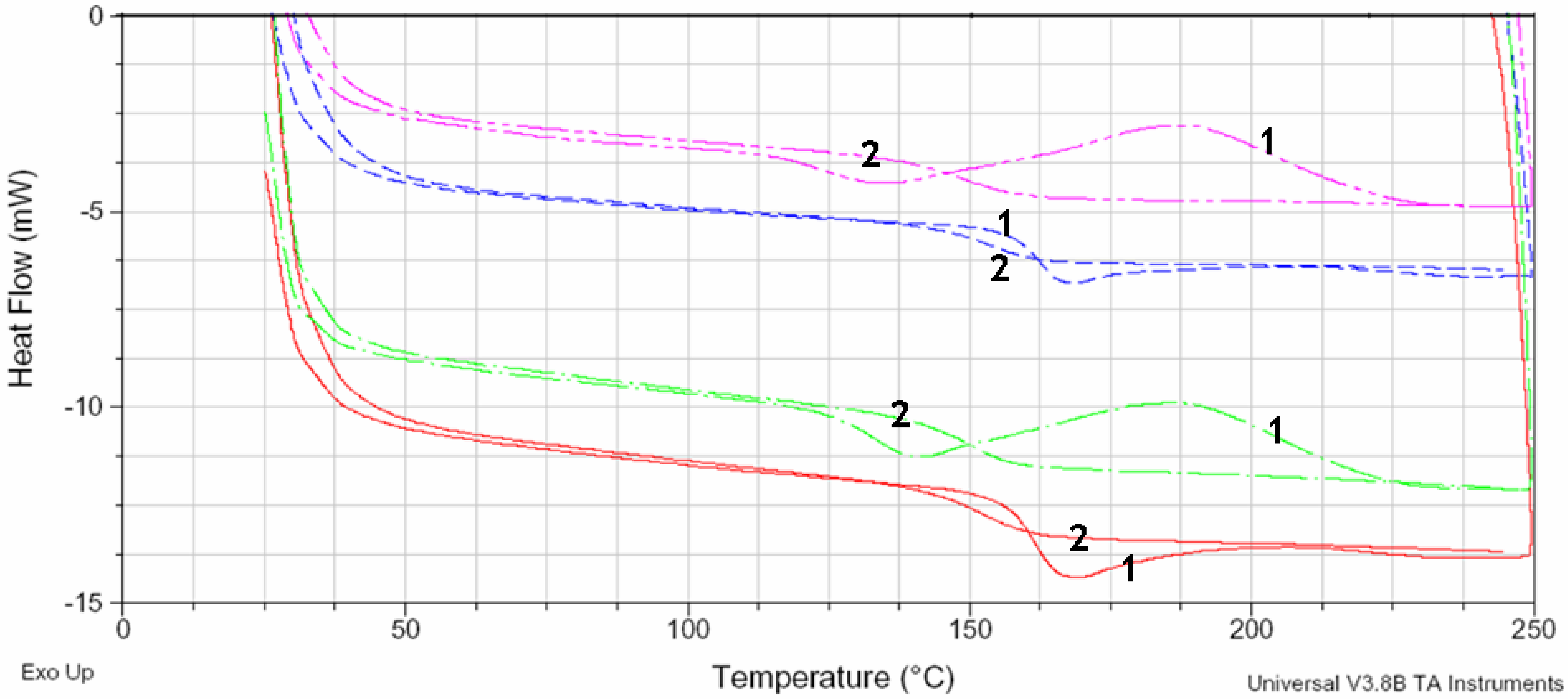
最終比較 Tg1 及 Tg2 二者, 依據以下規律判斷固化程度:

**IF 0 $^{O}$C =< Tg2 - Tg1 =< 5 $^{O}$C** 表示反應完全

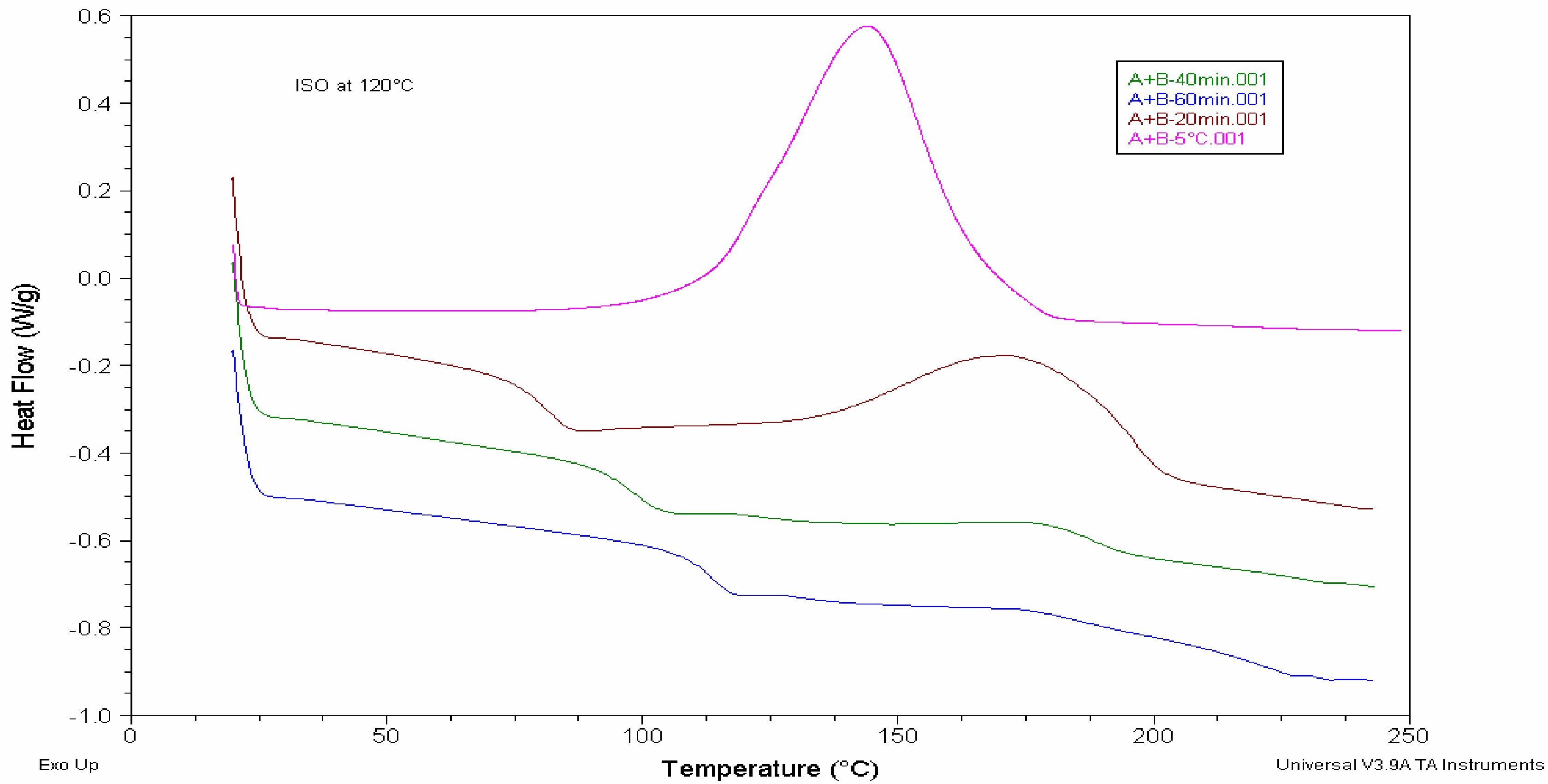
**IF Tg2 -Tg1 > 5 $^{O}$C** 表示反應不完全

**IF 0 $^{O}$C > Tg2 - Tg1** 表示**LED**耐熱性不佳

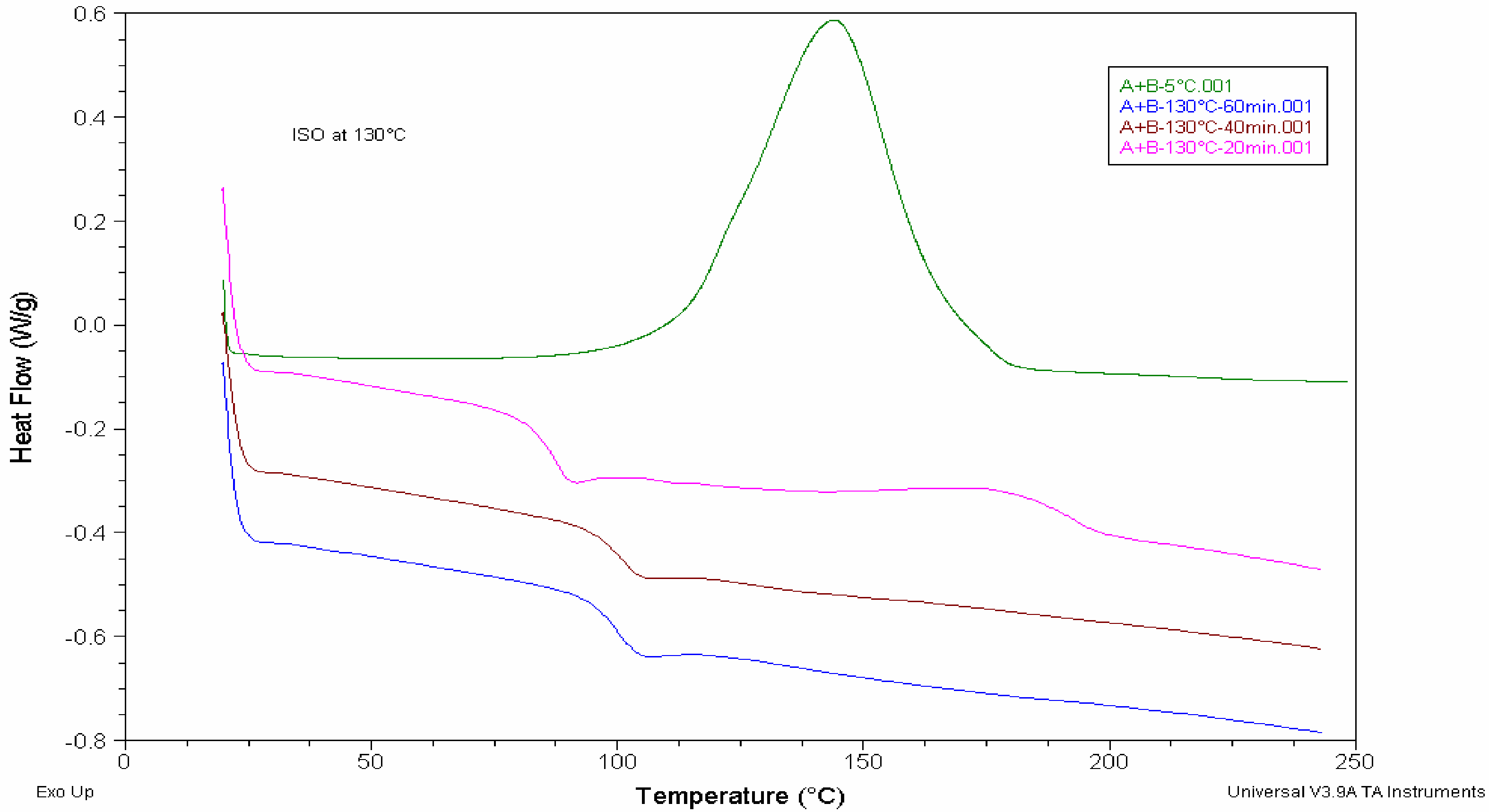
# 利用Tg1/Tg2的方式評價Epoxy是否完全固化?



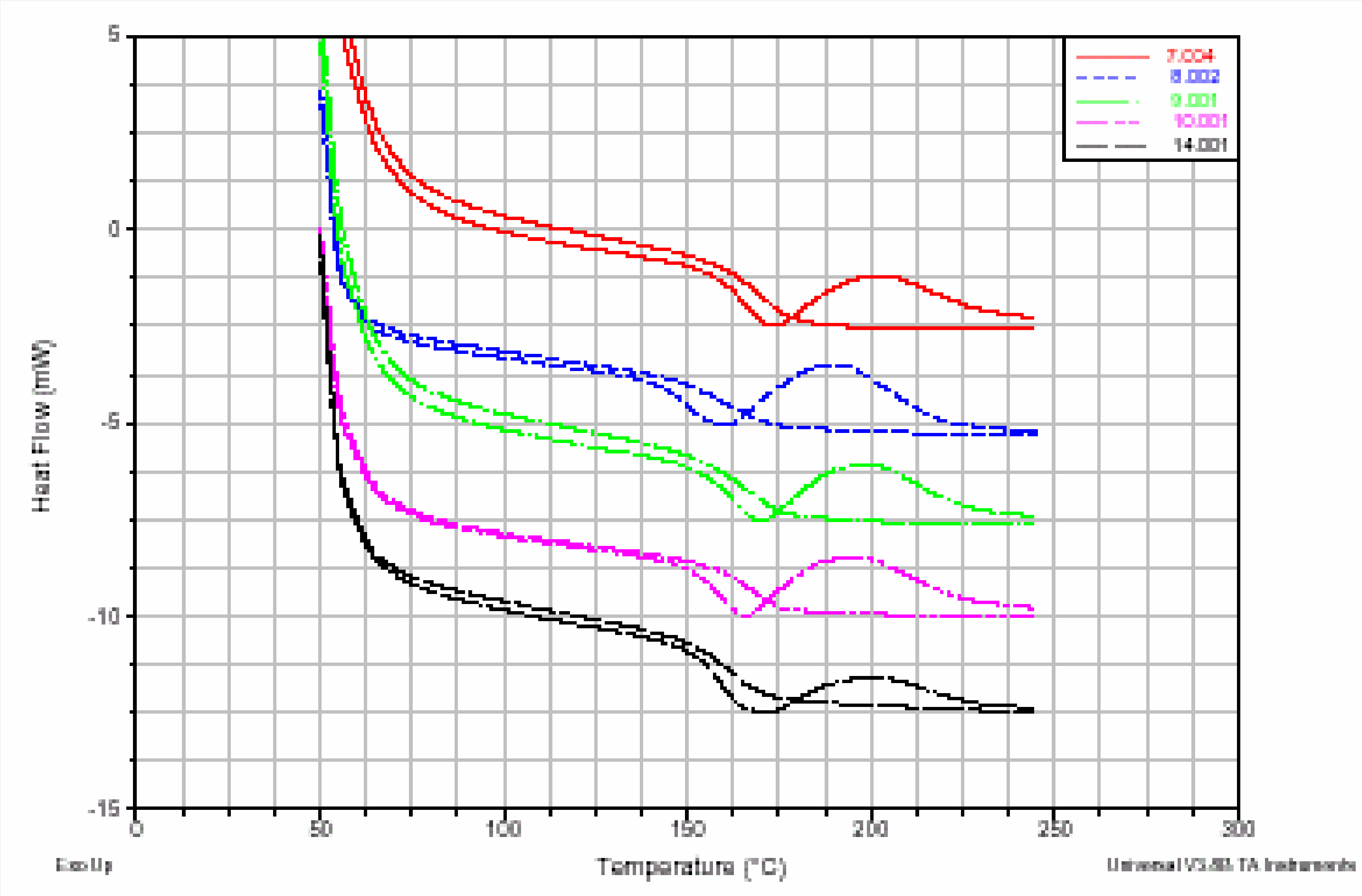
# DSC: Determination of % Cure



# DSC: Determination of % Cure

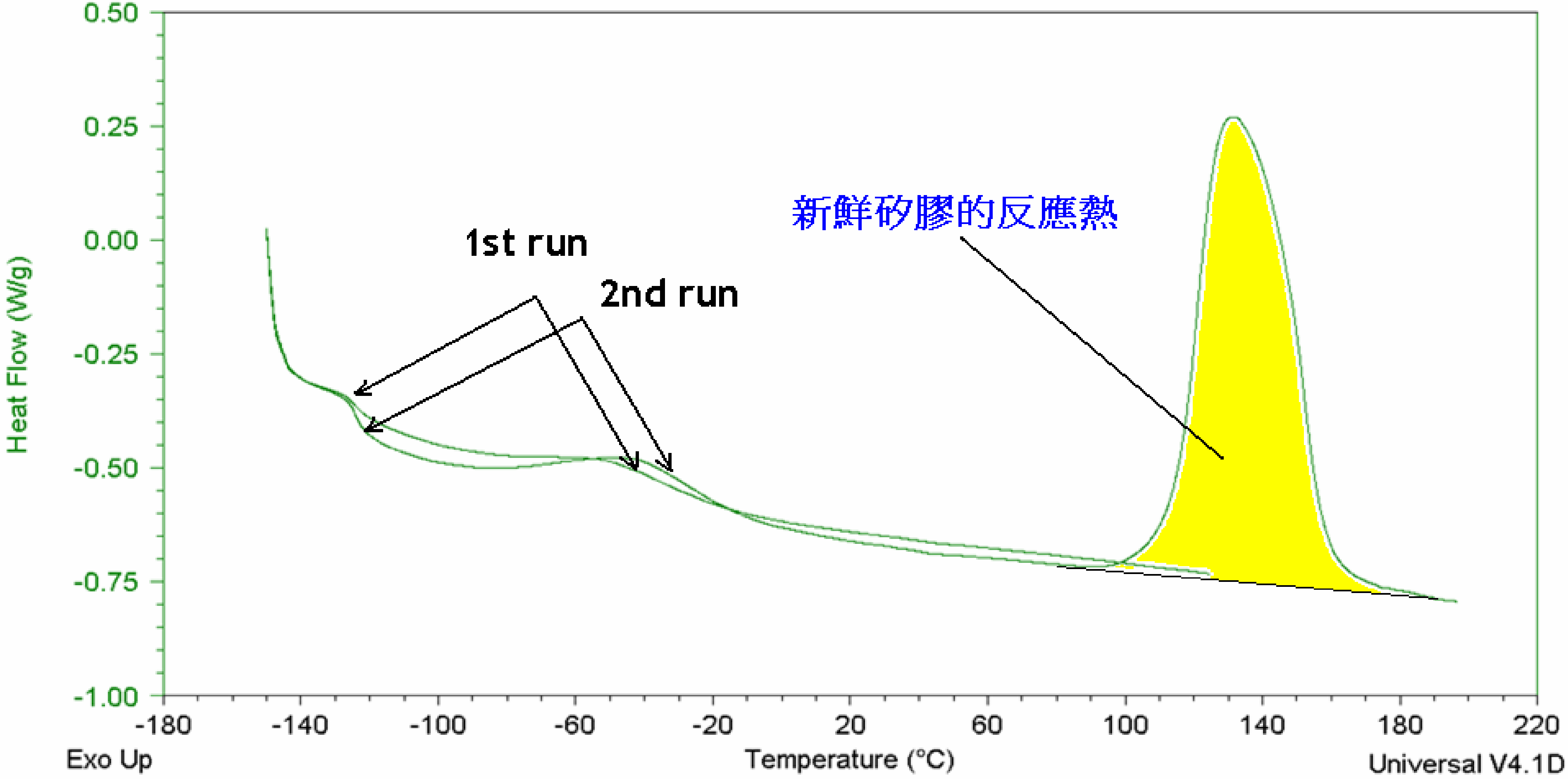


# DSC: SAME BEKING TEMP. DIFFERENT FURANCE

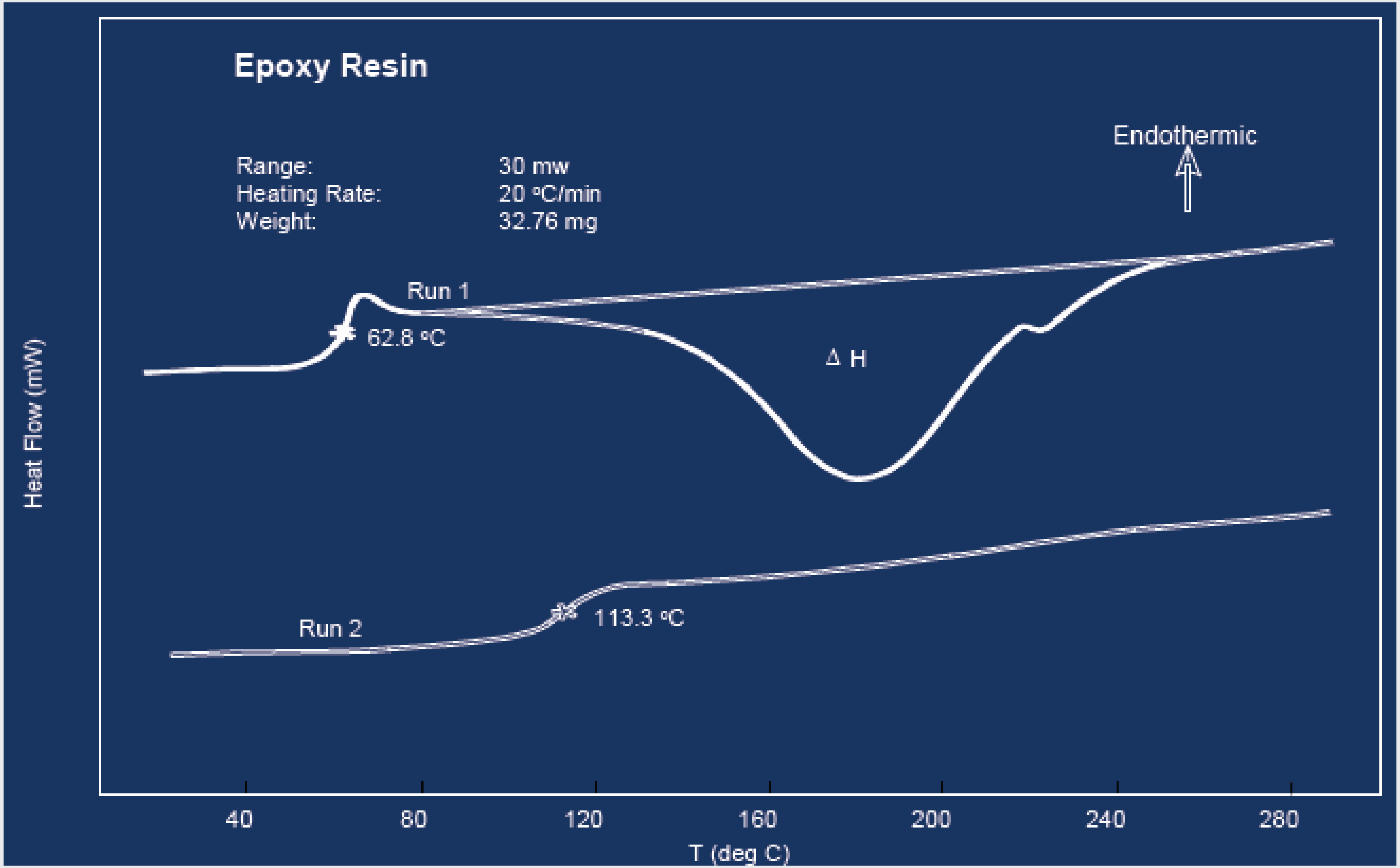


# 以DSC觀察矽膠封裝的行為

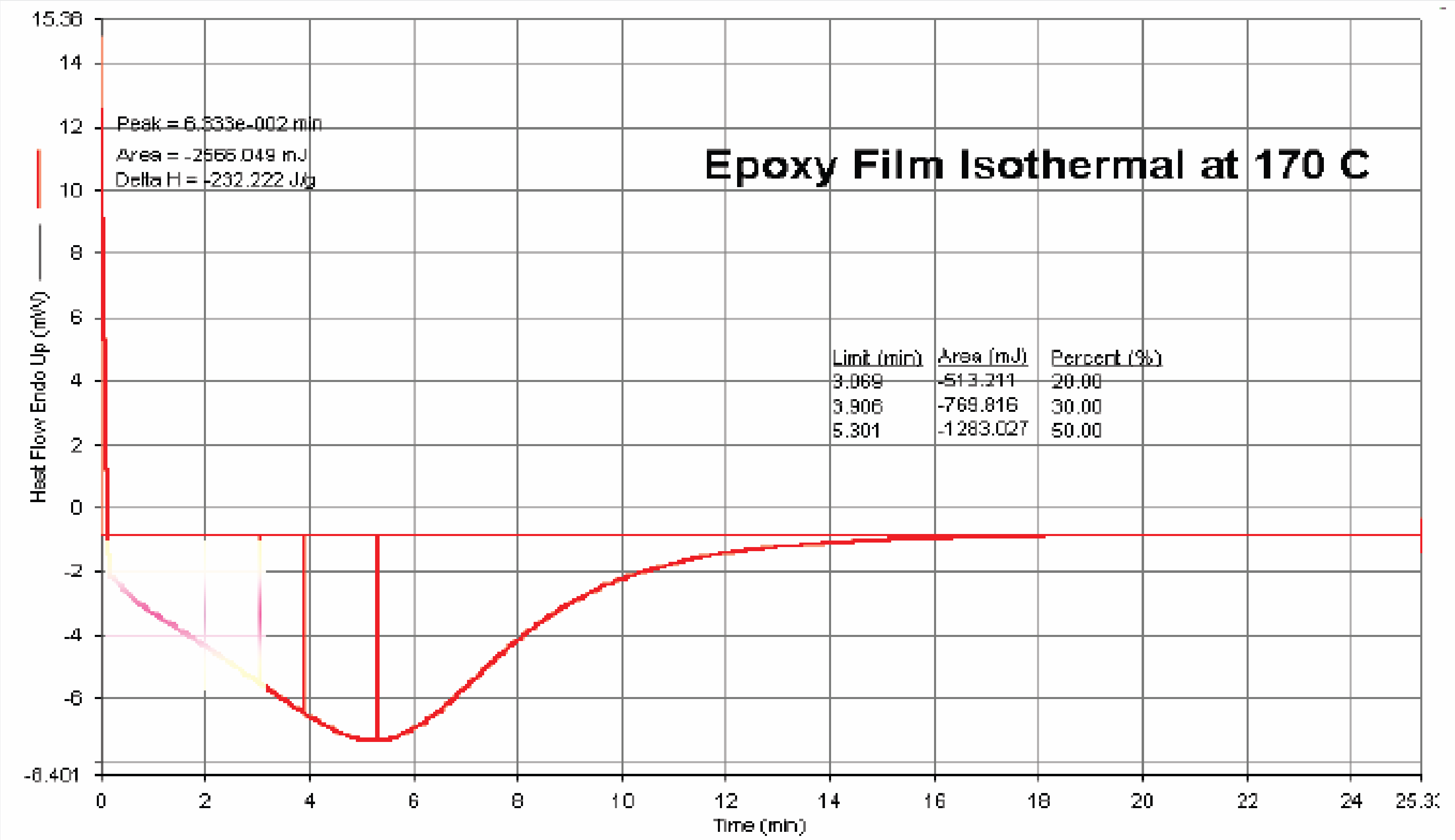
NETZSCH



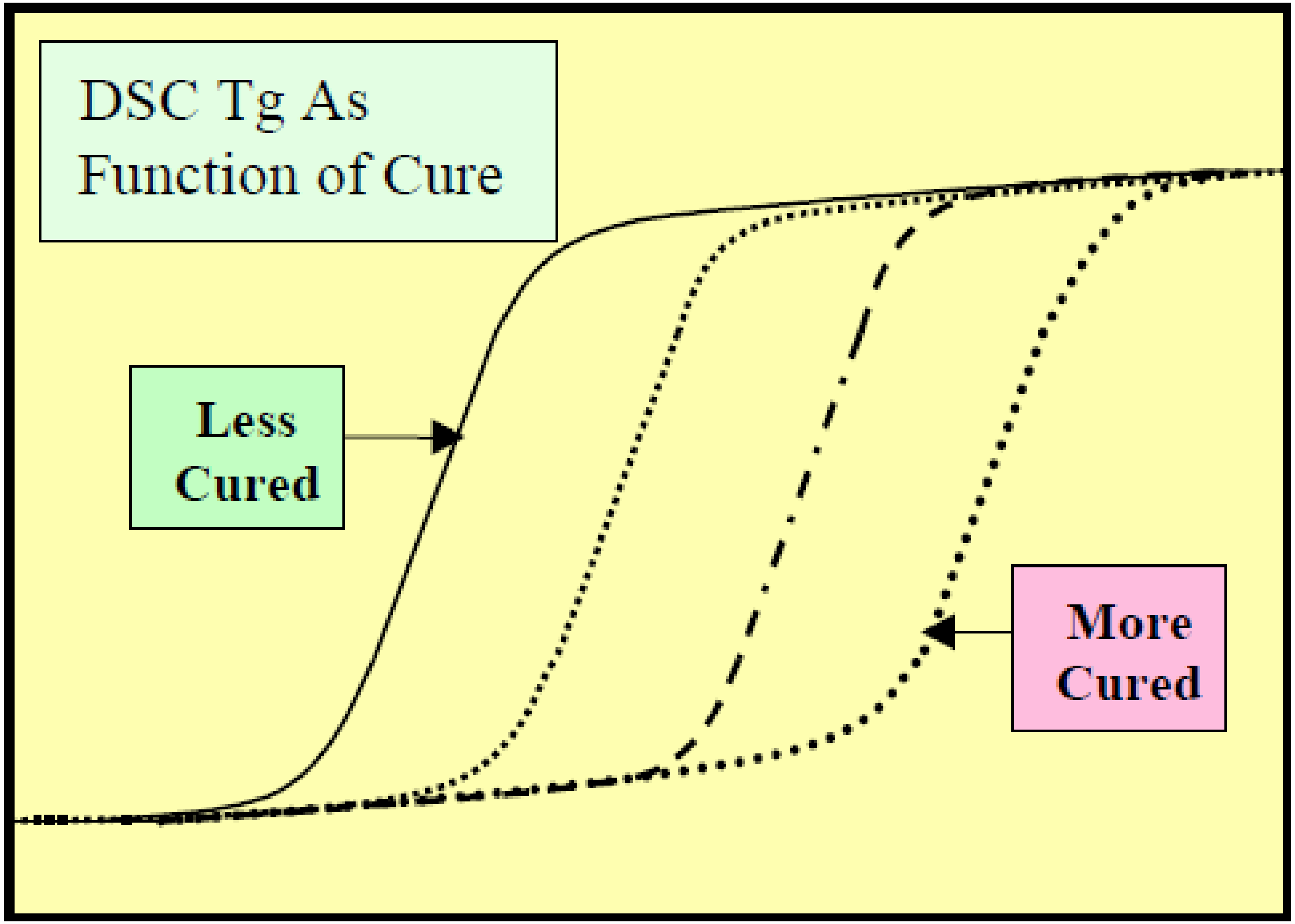
# *Cross-linking upon heating*



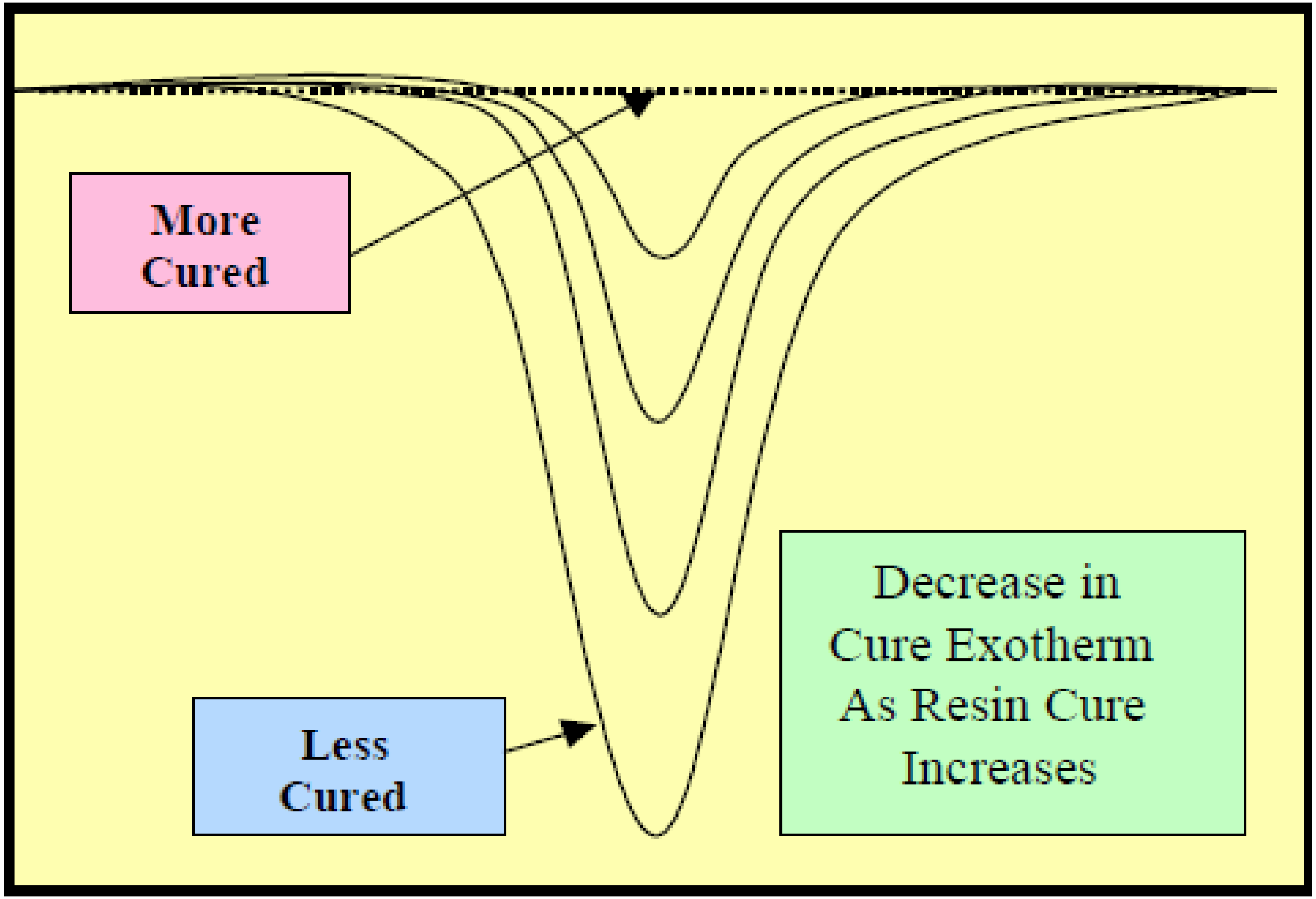
# *Cross-linking upon heating*



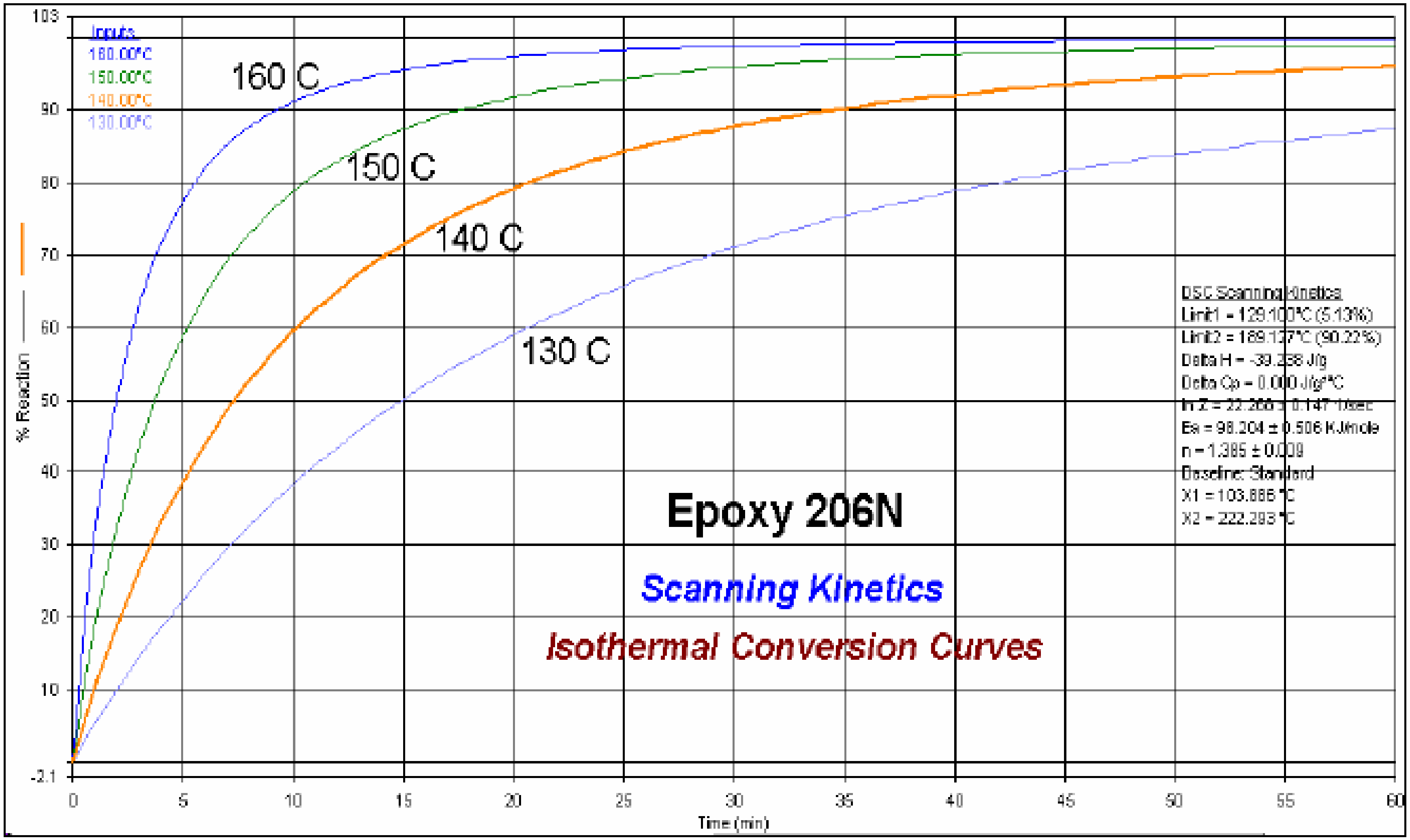
# *Degree of cross-linking*



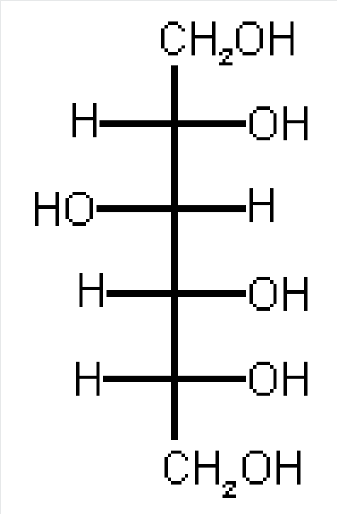
# *Degree of cross-linking*



# *動力學分析-- Scanning Kinetics(Option)*

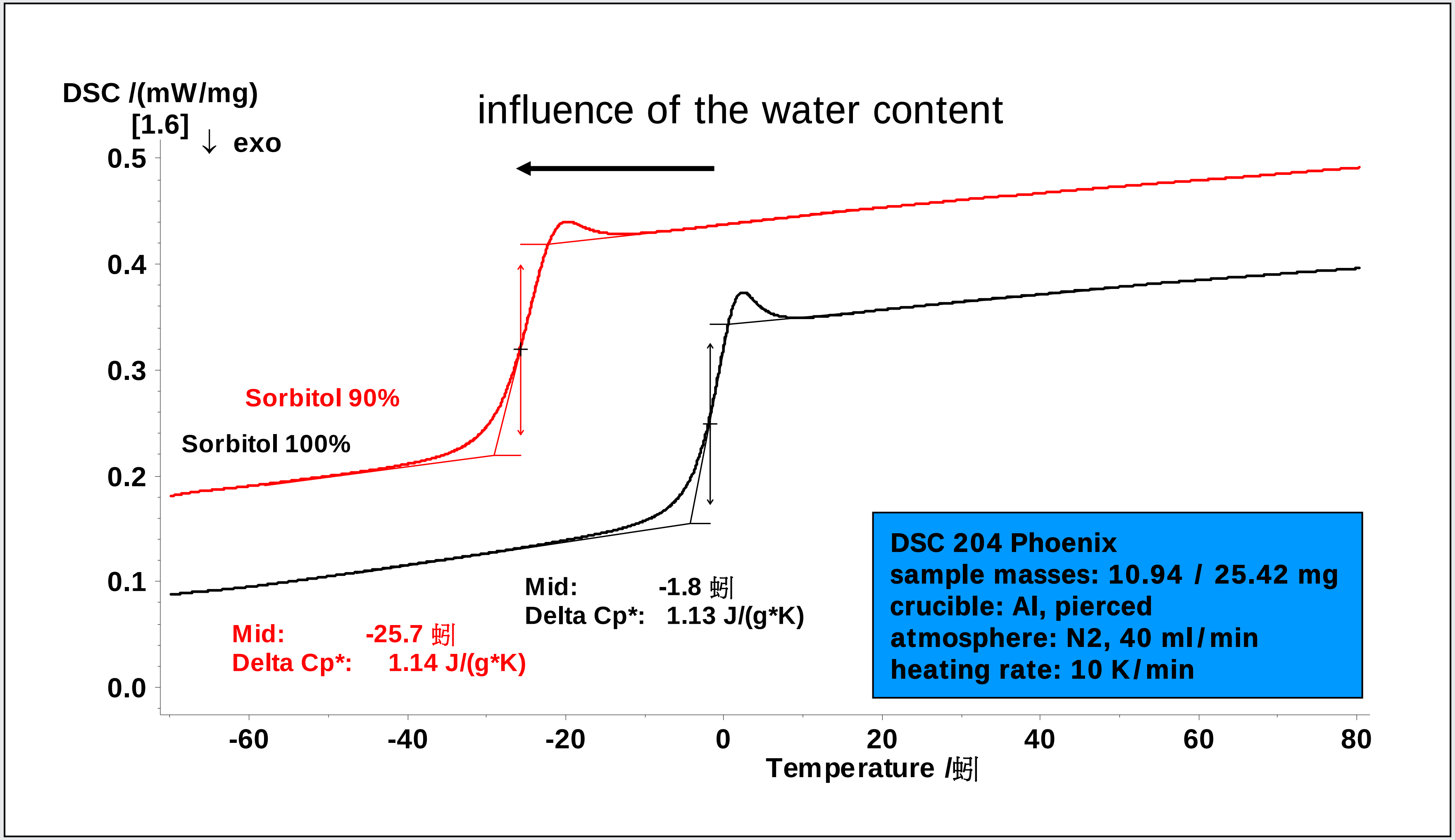


# Application: Sorbitol

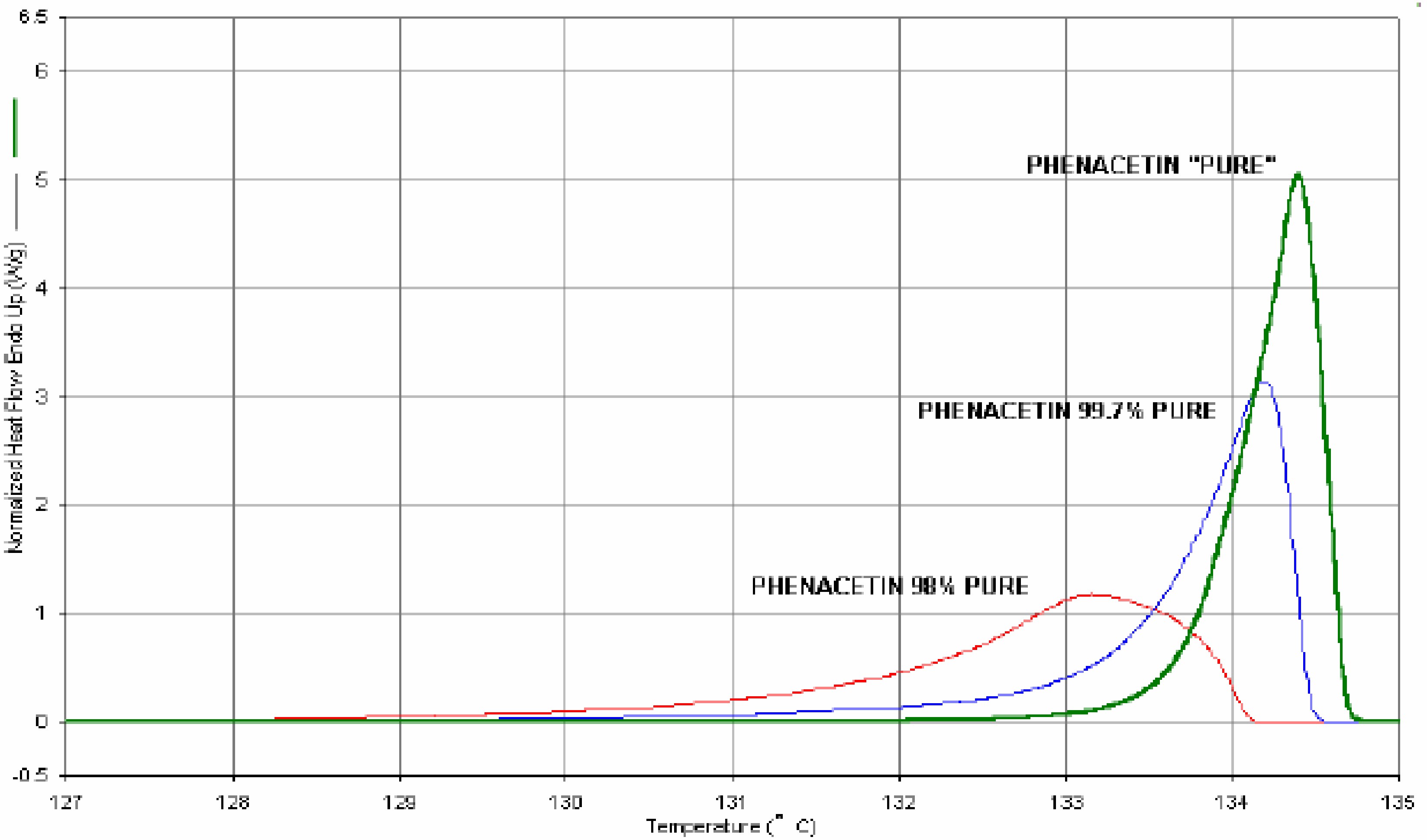




# Application: Sorbitol

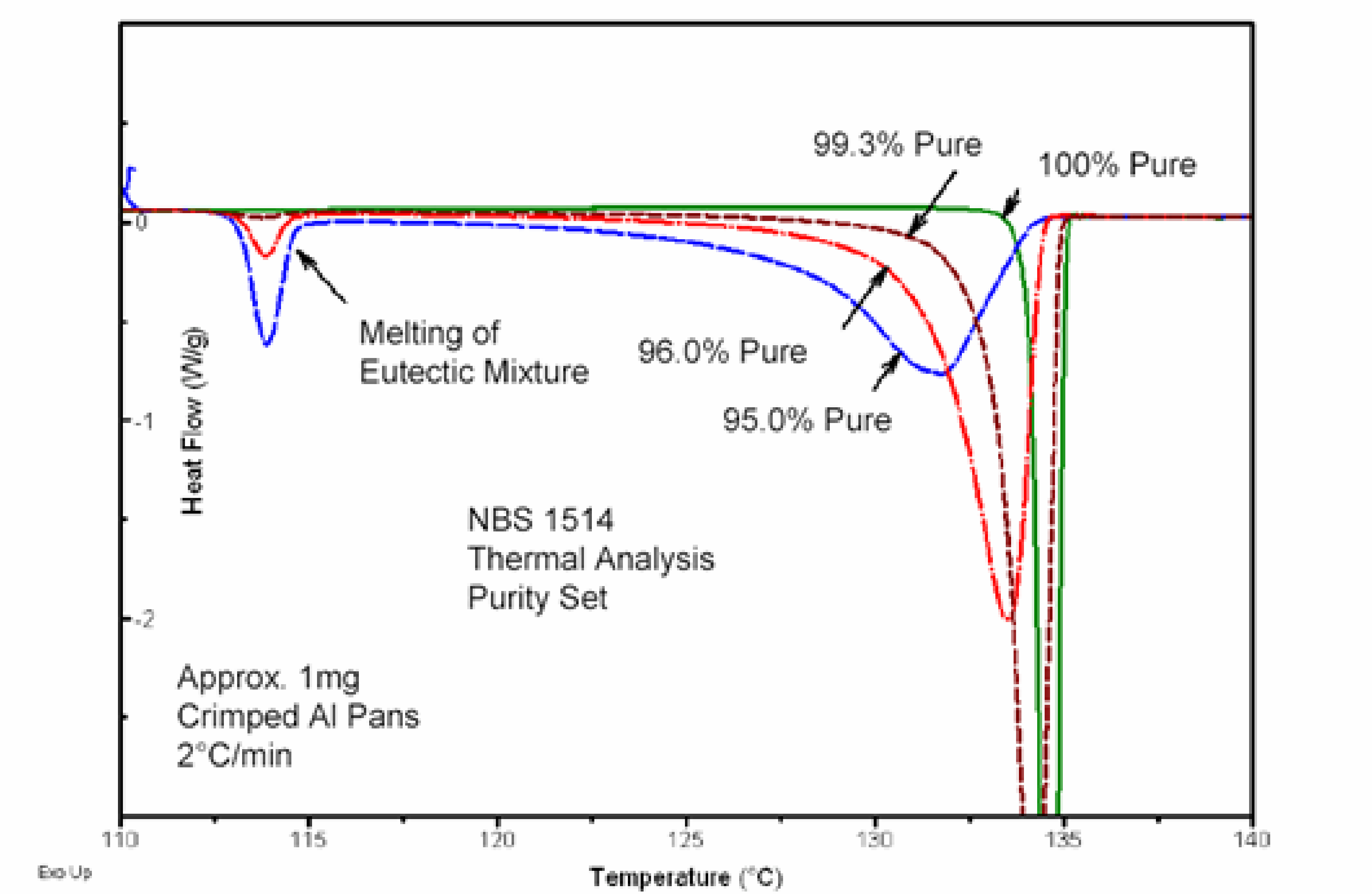


# *Determination of purity*

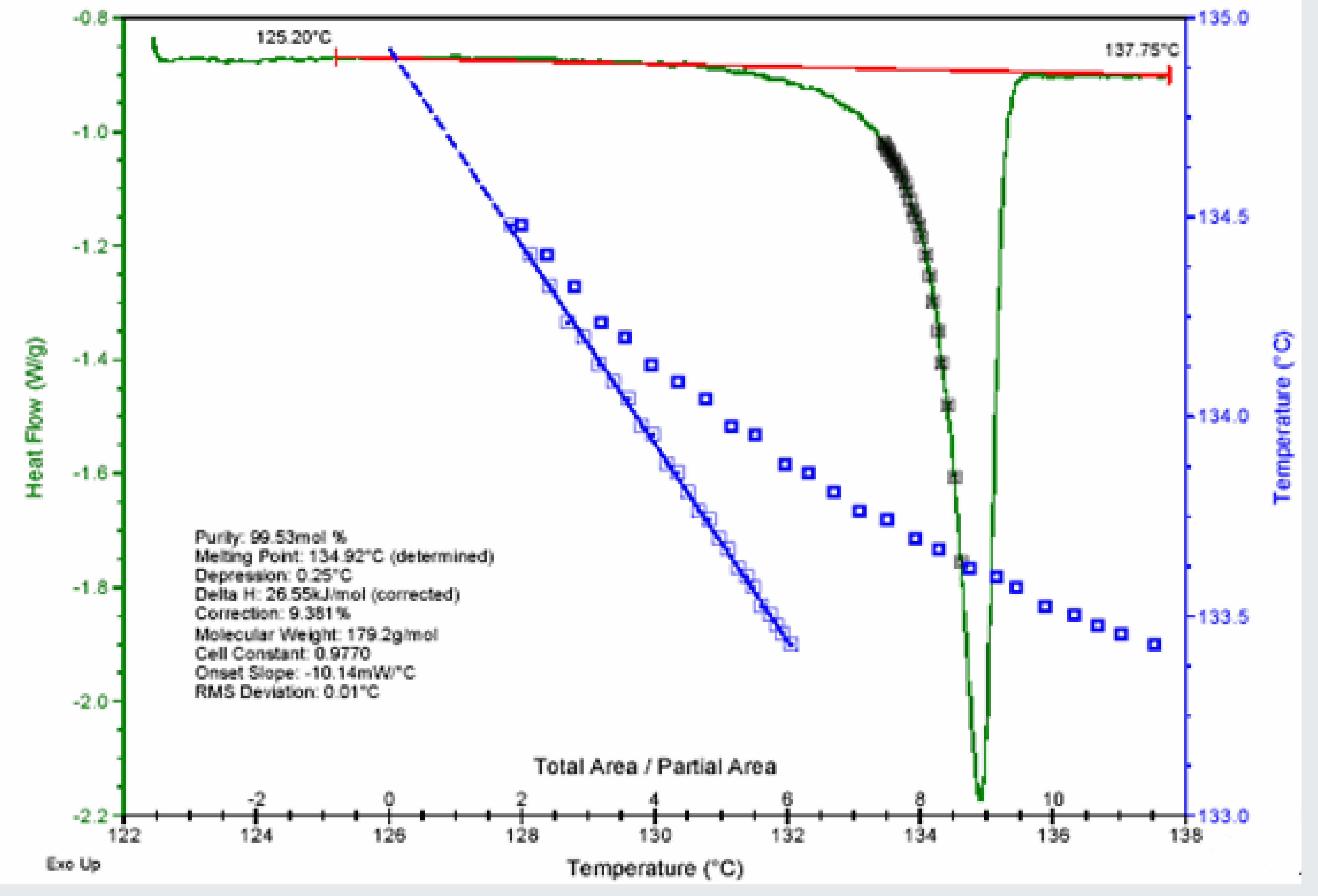



輸入分子量即可求出純度%

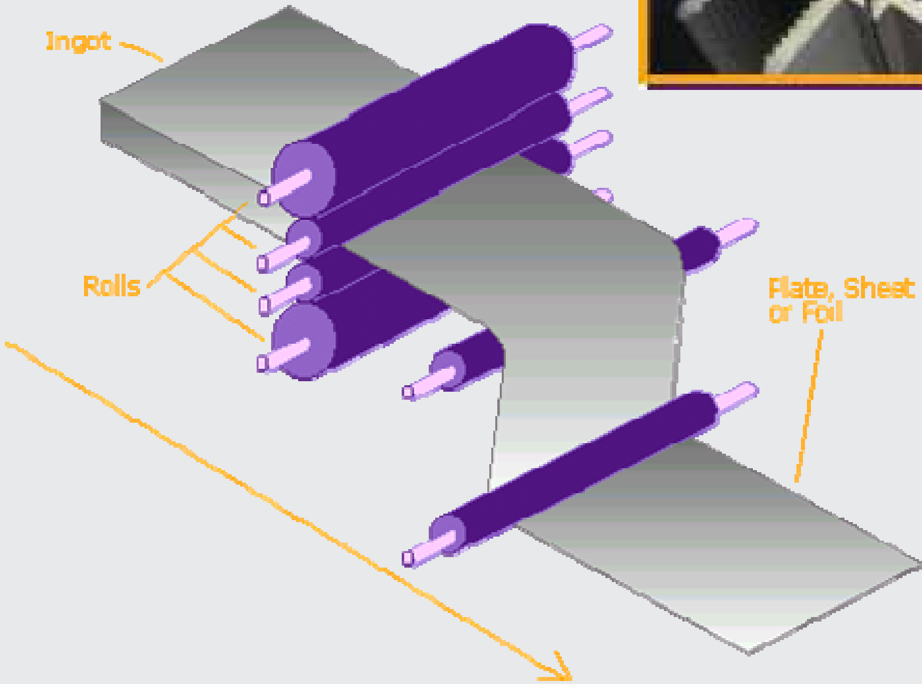
# 纯度对熔化的影响


99.3% Pure
100% Pure
Melting of
Eutectic Mixture
96.0% Pure
95.0% Pure
NBS 1514
Thermal Analysis
Purity Set
Approx. 1mg
Crimped Al Pans
2°C/min
Heat Flow (W/g)
0
-1
-2
110
115
120
125
130
135
140
Exo Up
Temperature (°C)

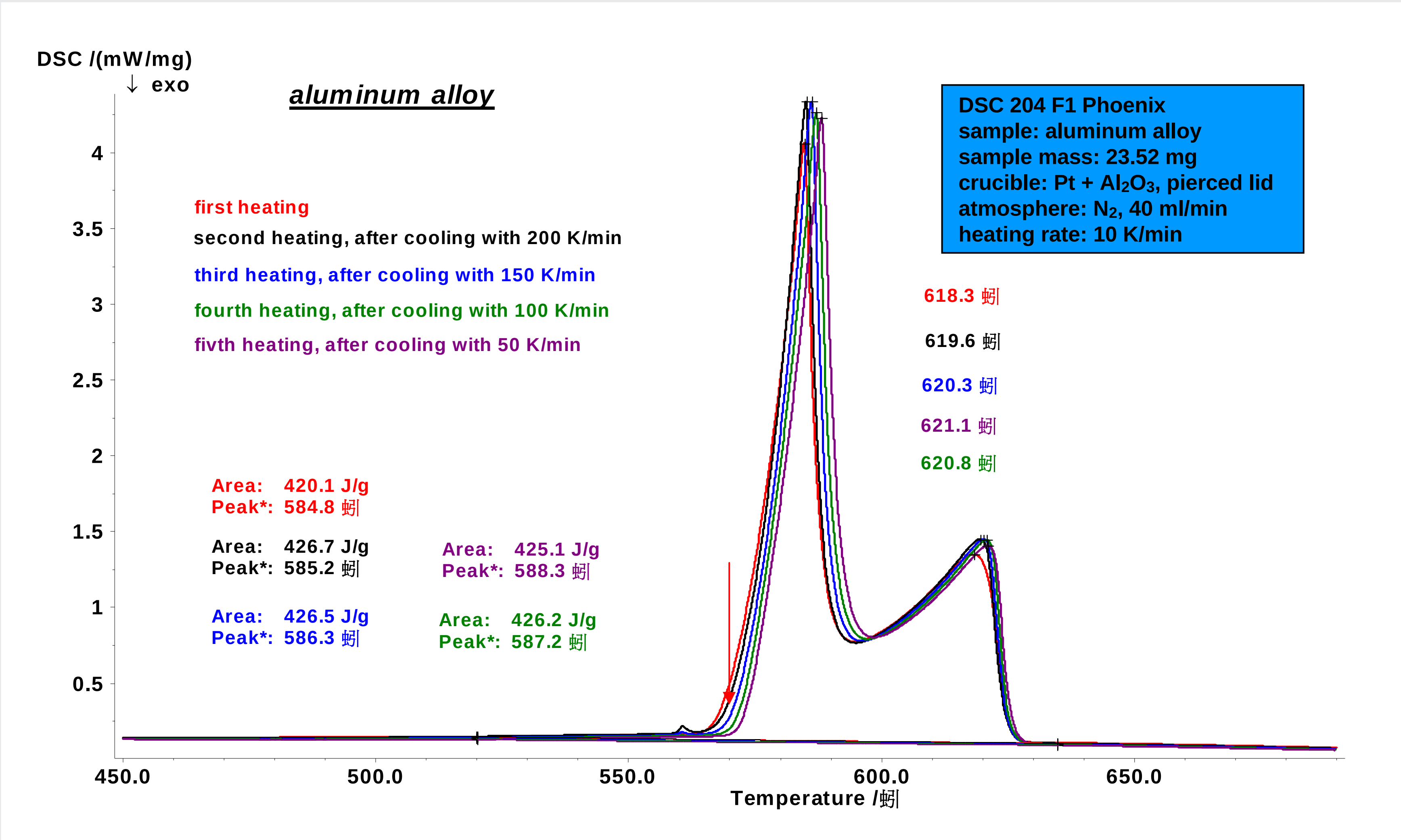
# Van‘t Hoff 纯度计算



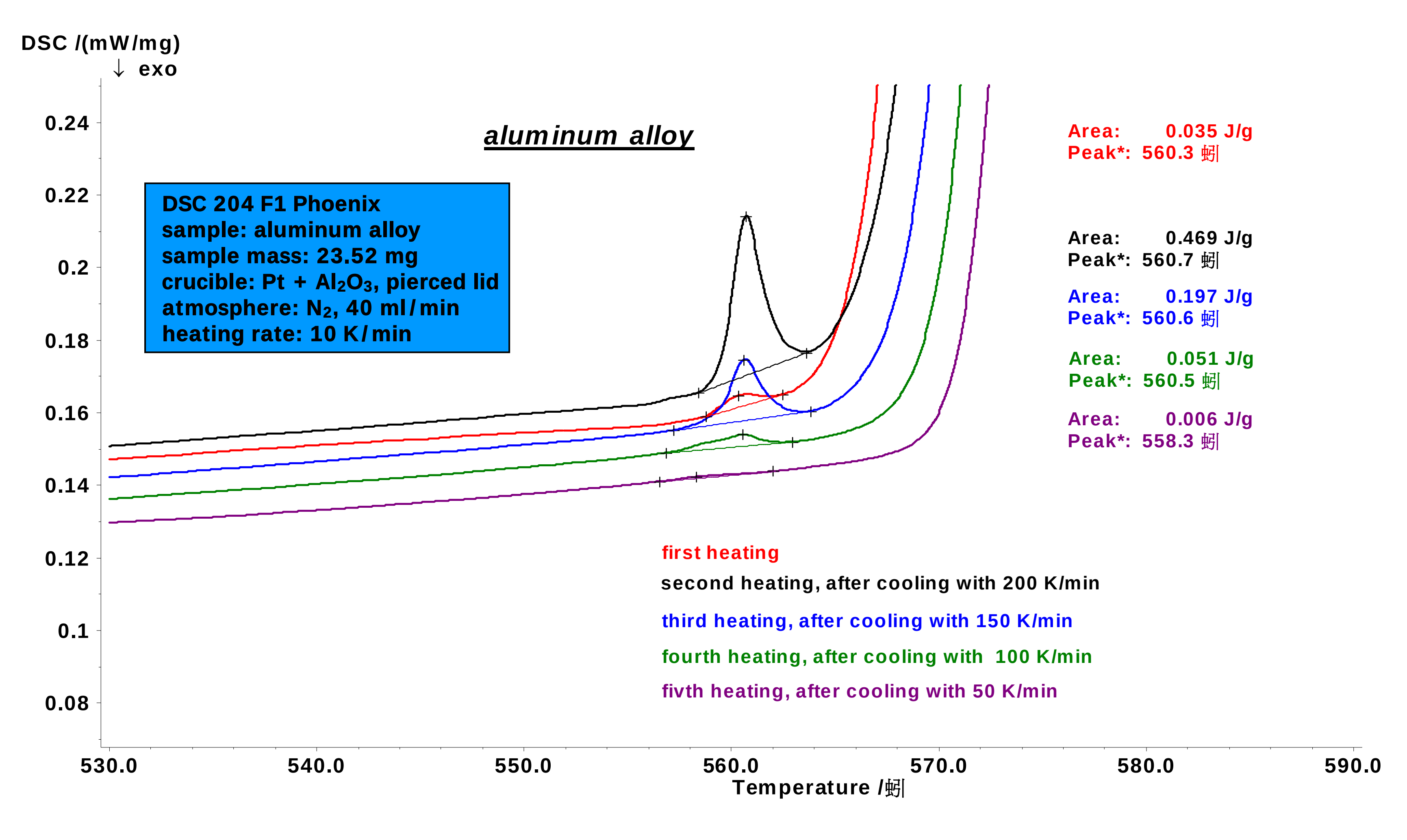
# Application: Melting/Solidifcation of AlSi-Alloys



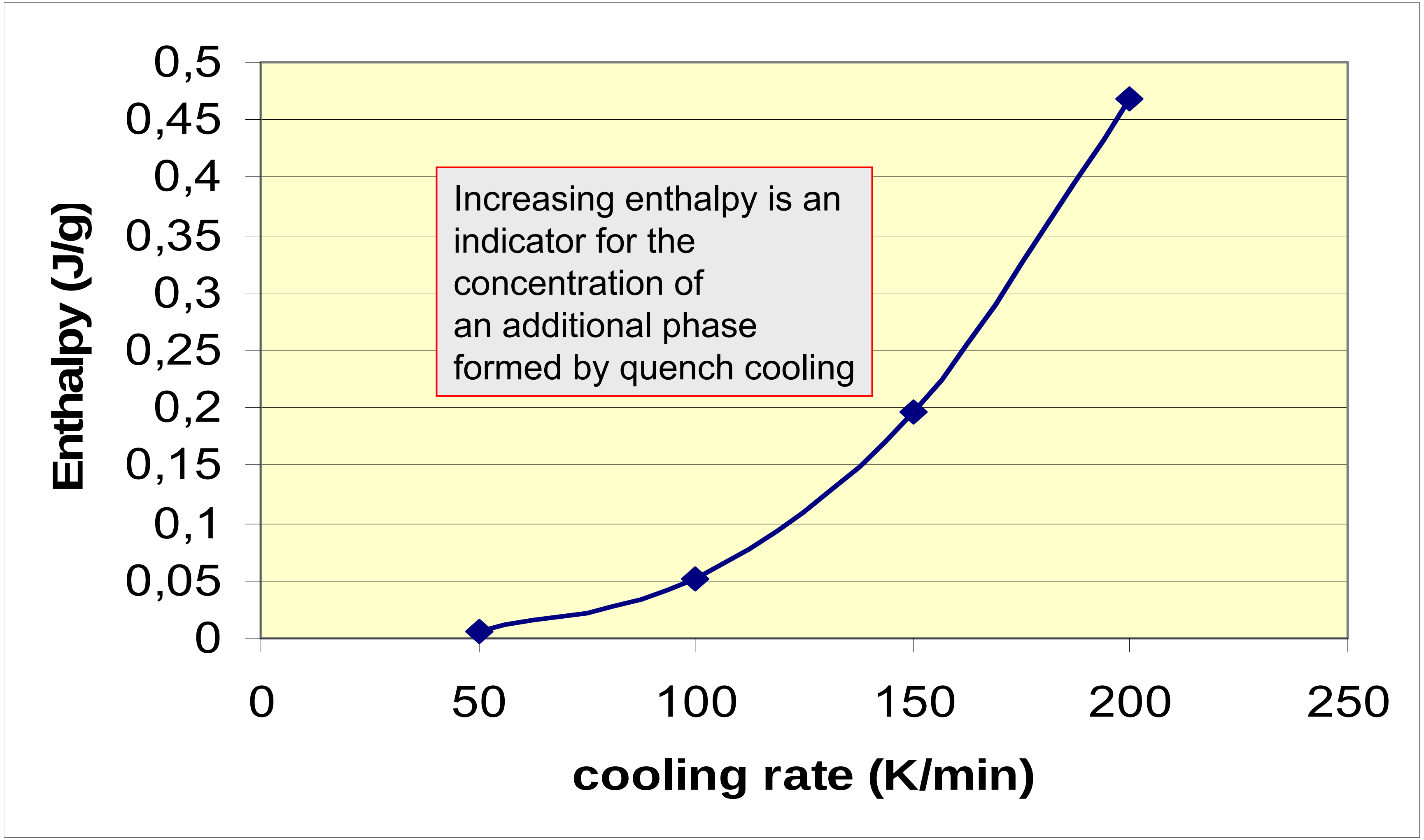
# Application: Melting/Solidifcation of AlSi-Alloys



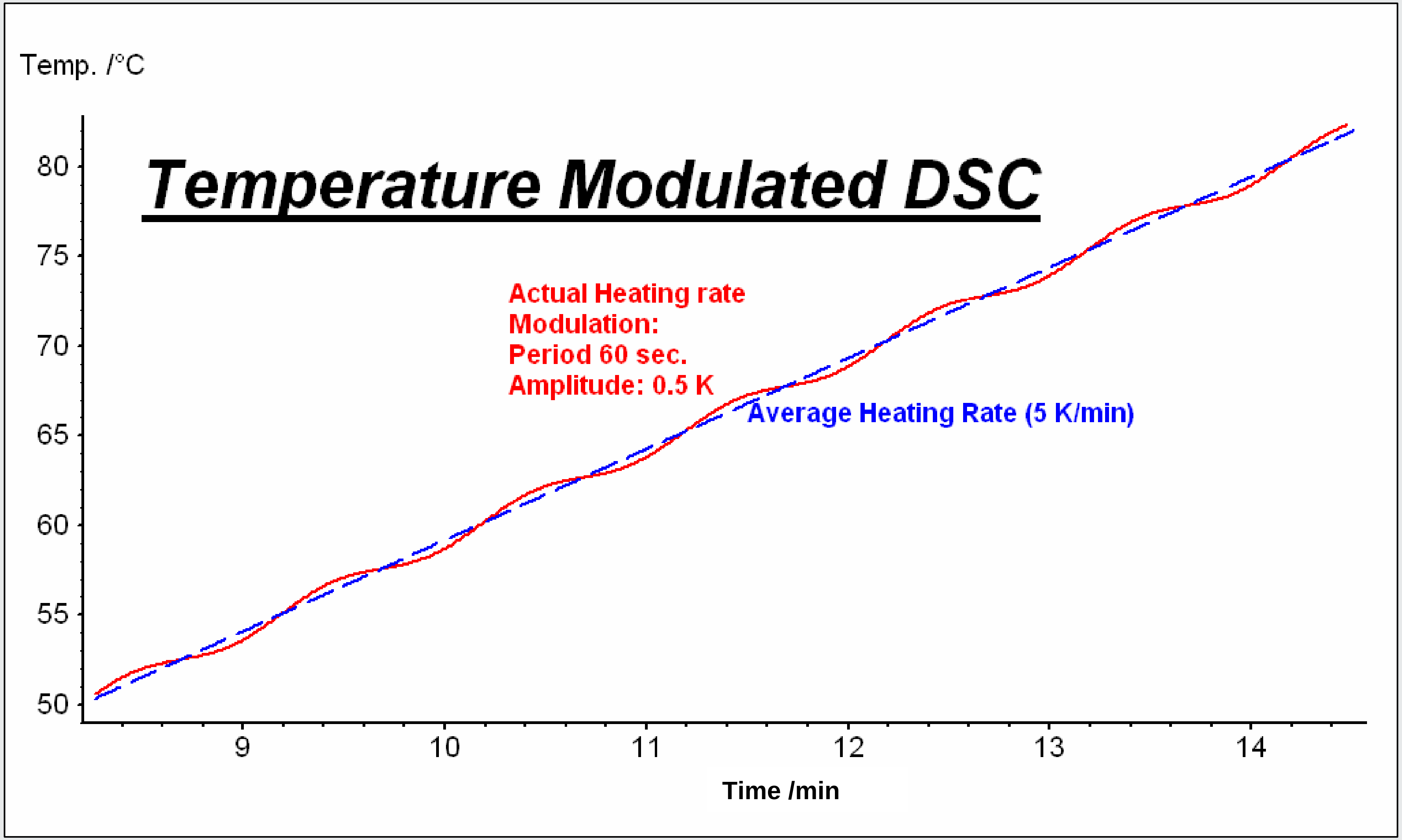
# Application: Melting/Solidification of AlSi-Alloys



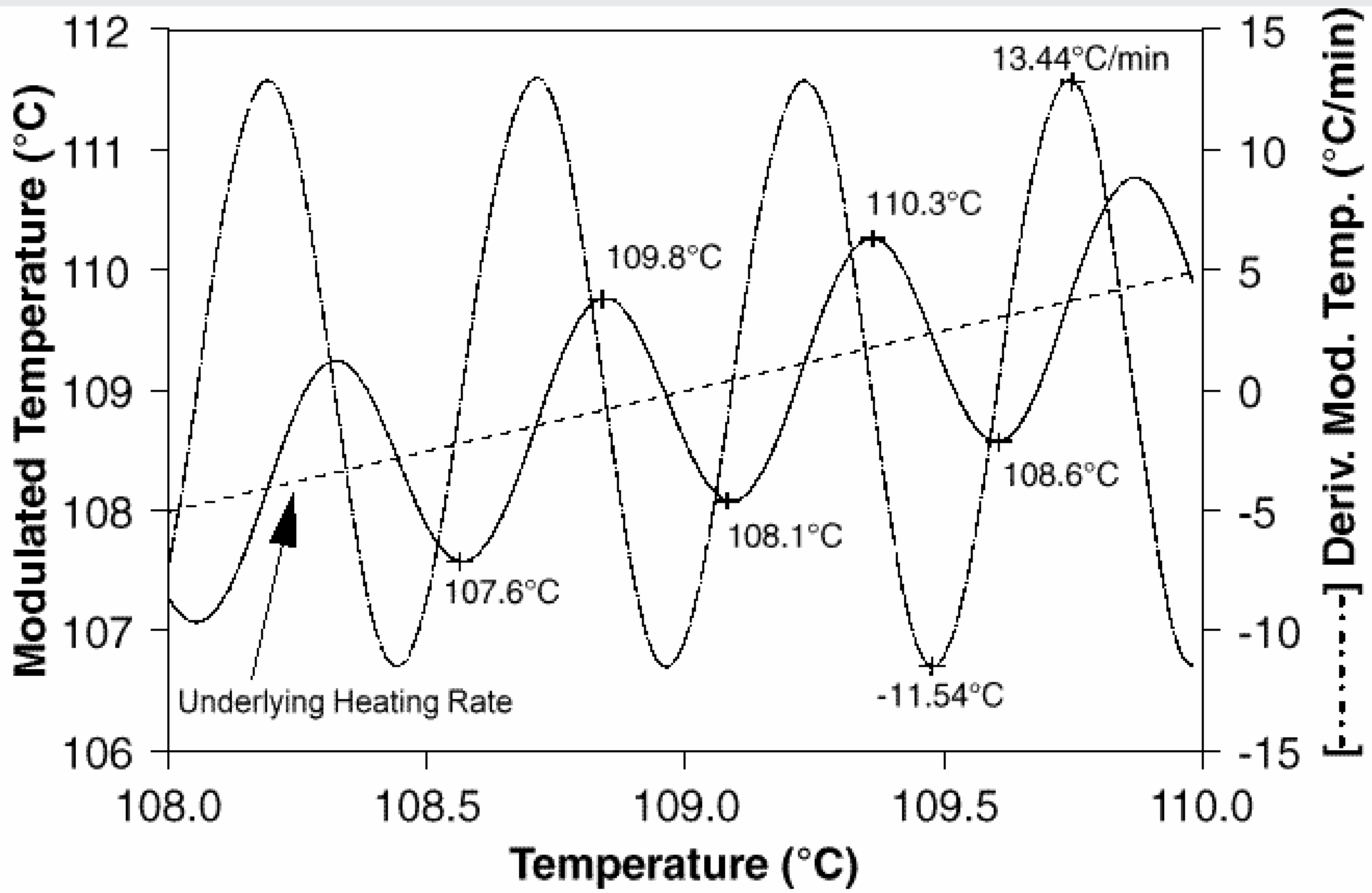
# Application: Melting/Solidification of AlSi-Alloys



# DSC 204 F1 – Temperature Modulation



# TM-DSC 的加熱曲線圖(循環的加熱/冷卻)



# 傳統DSC的限制

*基線彎曲及斜率改變影響微弱轉移偵測

*無法同時得到解析度與靈敏度

*無法測量絕對熱流改變,只能得到總熱流

*無法分辨及分析重疊的轉移(如玻璃化轉移及焓緩和或應力鬆弛)

*無法測量恒溫時比熱(Cp)改變

*比熱(Cp)測試繁瑣

*基線彎曲及斜率改變影響微弱轉移偵測

*無法準確量測初始結晶度

*無法直接測量熱傳導係數

# TM-DSC 主要的好處

- 同時得到高靈敏度及高解析度.
- 絕對熱流的測量
- 直接比熱測量(改進傳統DSC繁瑣測量方式).
- “擬恆溫”的條件下量測材料比熱的變化.
- “擬恆溫”的條件下量測材料冷結晶之現象.
- 將複雜的相轉移現象分解爲簡單易判斷的成份.
- 能夠決定材料之初始結晶度(ie.Initial Crystallinity).
- 消弭基線影響以取得微弱轉移溫度的測量.
- 解決傳統DSC所無法測試及解釋的現象
- 量測熱傳導率.

# TM-DSC 如何不同於DSC ？

| | DSC | TM-DSC | COMMENTS* |
|---|---|---|---|
| 實驗參數需要編輯那些? | 線性升溫速率 | 線性升溫速率 | TM-DSC的範圍由恆溫 -10 ºC/min |
| | | 溫度的振盪幅度 | 範圍由+/-0.01-10ºC/min |
| | | 溫度的振盪周期 | 範圍由+/-0.01-10ºC/min |

# TM-DSC 如何不同於DSC ?

| | DSC | TM-DSC | COMMENTS |
|---|---|---|---|
| 可獲得那些資訊? | 總熱流 | 振盪熱流 | 兩者都包含了樣品在測試其間所有發生的熱事件資訊. |
| | | 總熱流 | 在相同的平均加熱速率MDSC 與DSC的總熱流訊號是相同. |
| | | 可逆熱流 | 屬於總熱流中的比熱成分 |
| | | 不逆熱流 | 屬於總熱流中的動力成分 |
| | | 比熱 | |

# 瞬間熱流改變(DSC vs TM-DSC)

## DSC

$$dQ/dt = Cp\,(\beta) + f\,(t,T)$$

where: $dQ/dt$ = 總熱流

Cp = 比熱

T = 絕對溫度

$Cp\,(\beta\,)$ = 比熱成份

$f\,(t,T)$ = 動力成份

## TM-DSC

$$dQ/dt = Cp\,(\beta + AT\,\omega\,\cos\omega t) + A_k(\sin\,\omega t) + f(T,t)$$

where $(\beta + AT\,\omega\,\cos\omega t)$ = 測到的加熱速率 (dT/dt)

AK = 振幅對溫度振盪的動力成分回應

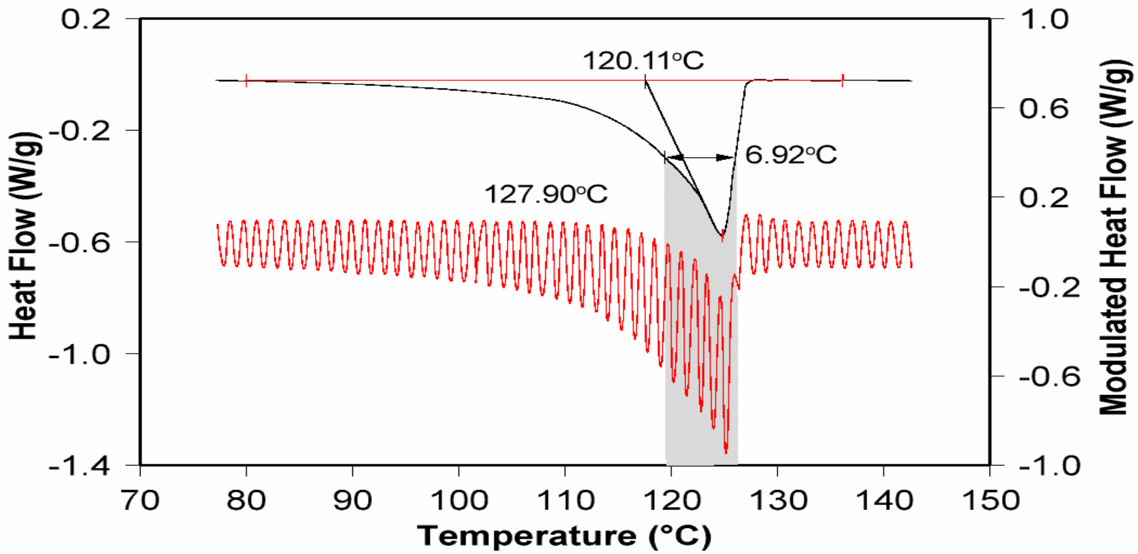
$f\,(T,t)$ = 在沒有溫度振盪的情形下的動力成分回應

# TM-DSC 訊號的計算

綜論：

所有的TM-DSC之訊號是從以下三個基本量測訊號獲得的.

- 時間(Time)
- 振盪熱流(Modulated Heat Flow)
- 振盪溫度(Modulated Temperature)




$$\beta = \frac{T_{1/2}}{n\ P}\ 60s/min$$

Where:

$T_{1/2}$ = full width at helf height

n = number of cycles

P = period

# TM-DSC - 瞬間熱流改變

## TM-DSC

dQ/dt = Cp (β + AT ω cosωt) + $A_k$(sin ωt) +f(T,t)

= Reversing heating flow
玻璃轉移
熔點(some)
比熱

+ Nonreversing heat flow
焓松弛
氣化,蒸發
冷結晶
裂解
固化或熟化
熔點(some)

Where (β + AT ω cosωt) = 測到的加熱速率 (dT/dt)
AK = 振幅對溫度振盪的動力成分回應
f (T,t)= 在沒有溫度振盪的情形下的動力成分回應

# TM-DSC 訊號 - 總熱流

總熱流是振盪熱流的平均值,這結果與傳統DSC在相同平均升溫速率下的實驗值相同.

定義 ： 樣品的所有熱事件的總和.

計算 ： 利用傅利葉轉換的方法連續的將振盪熱流算出平均值.

# TM-DSC 訊號 - 可逆熱流(比熱成份)

可逆熱流是總熱流的比熱成份,將量測的比熱乘以平均加熱速率計算而得.

可逆熱流 = -Cp x平均加熱速率

<u>計算的基礎</u>

$$dQ/dt = Cp\beta + f(T,t)$$

where dQ/dt = 總熱流

Cp = Cp量測值

β = 平均加熱速率

Cpβ = 總熱流的比熱成份(可逆的)

f (T,t) =總熱流的動力成份(不可逆的)

# TM-DSC 訊號 - 不可逆熱流(動力成份)

不可逆熱流是總熱流的動力成份,係由總熱流扣除可逆熱流計算而得.

不可逆熱流 = 總熱流 - 可逆熱流

= dQ/dt - $Cp\beta$

計算的基礎

$$dQ/dt = Cp\beta + f(T,t)$$

where dQ/dt = 總熱流

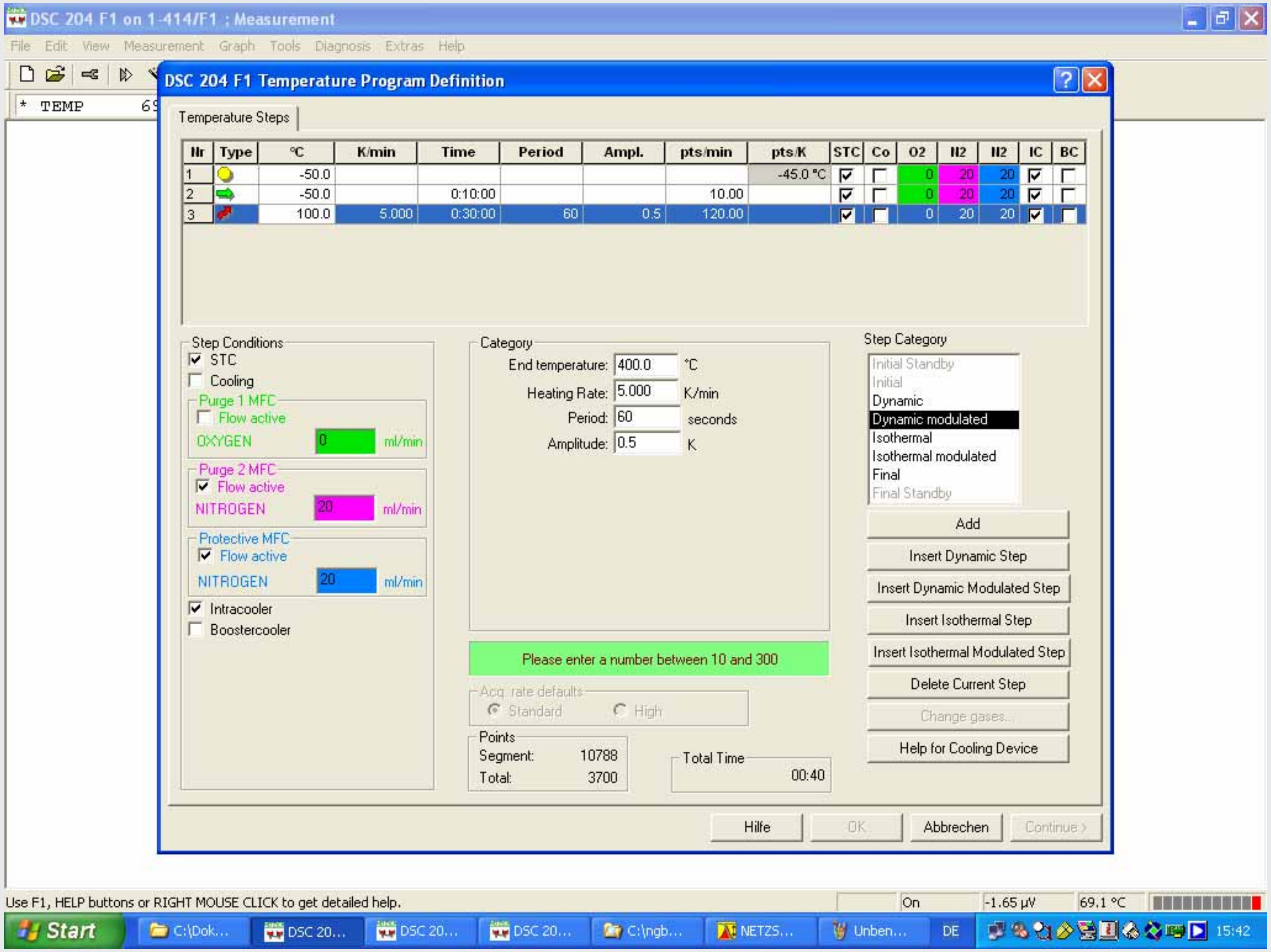
Cp = Cp量測值

$\beta$ = 平均加熱速率

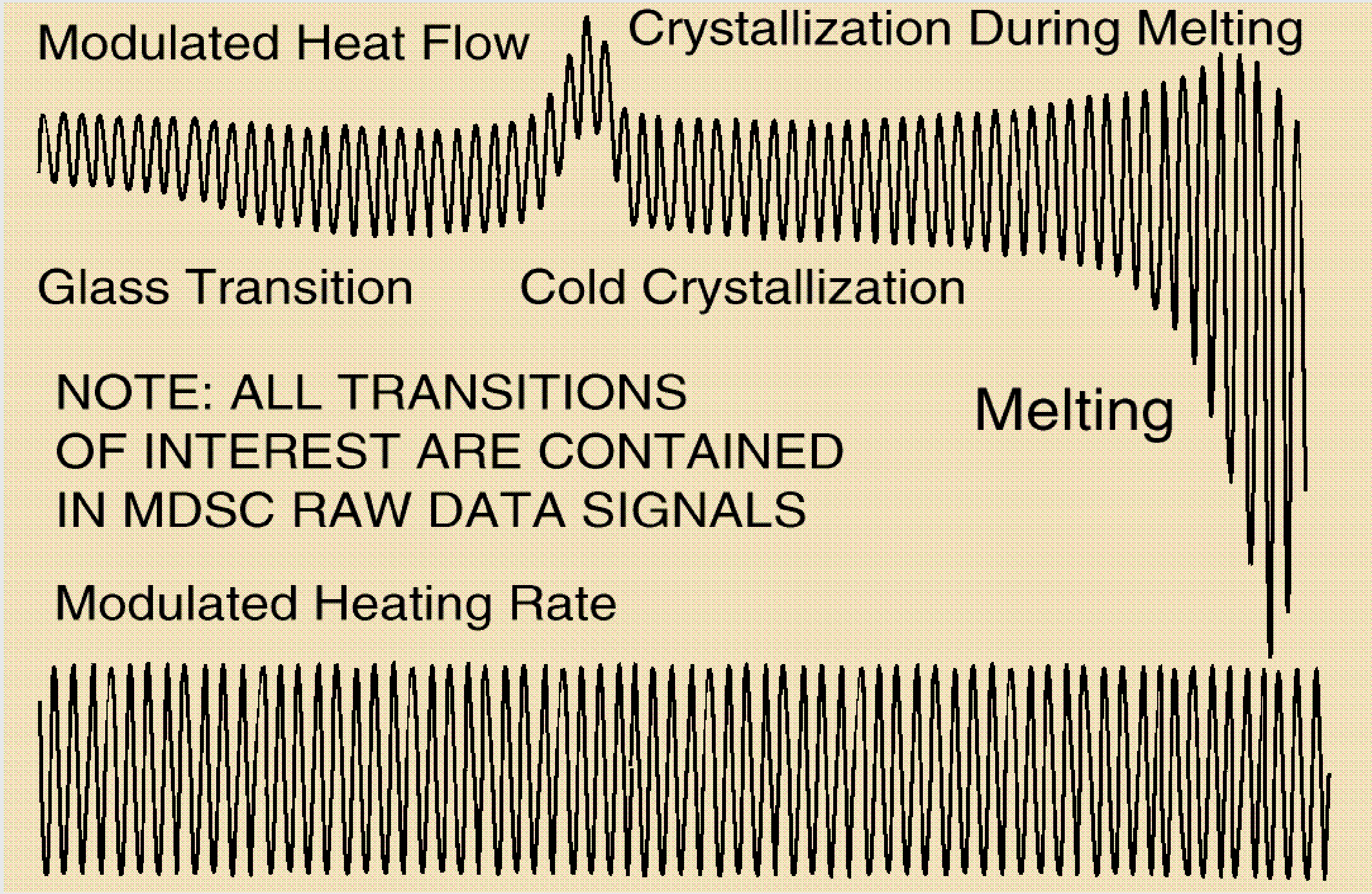
$Cp\beta$ = 總熱流的比熱成份(可逆的)

f (T,t) =總熱流的動力成份(不可逆的)

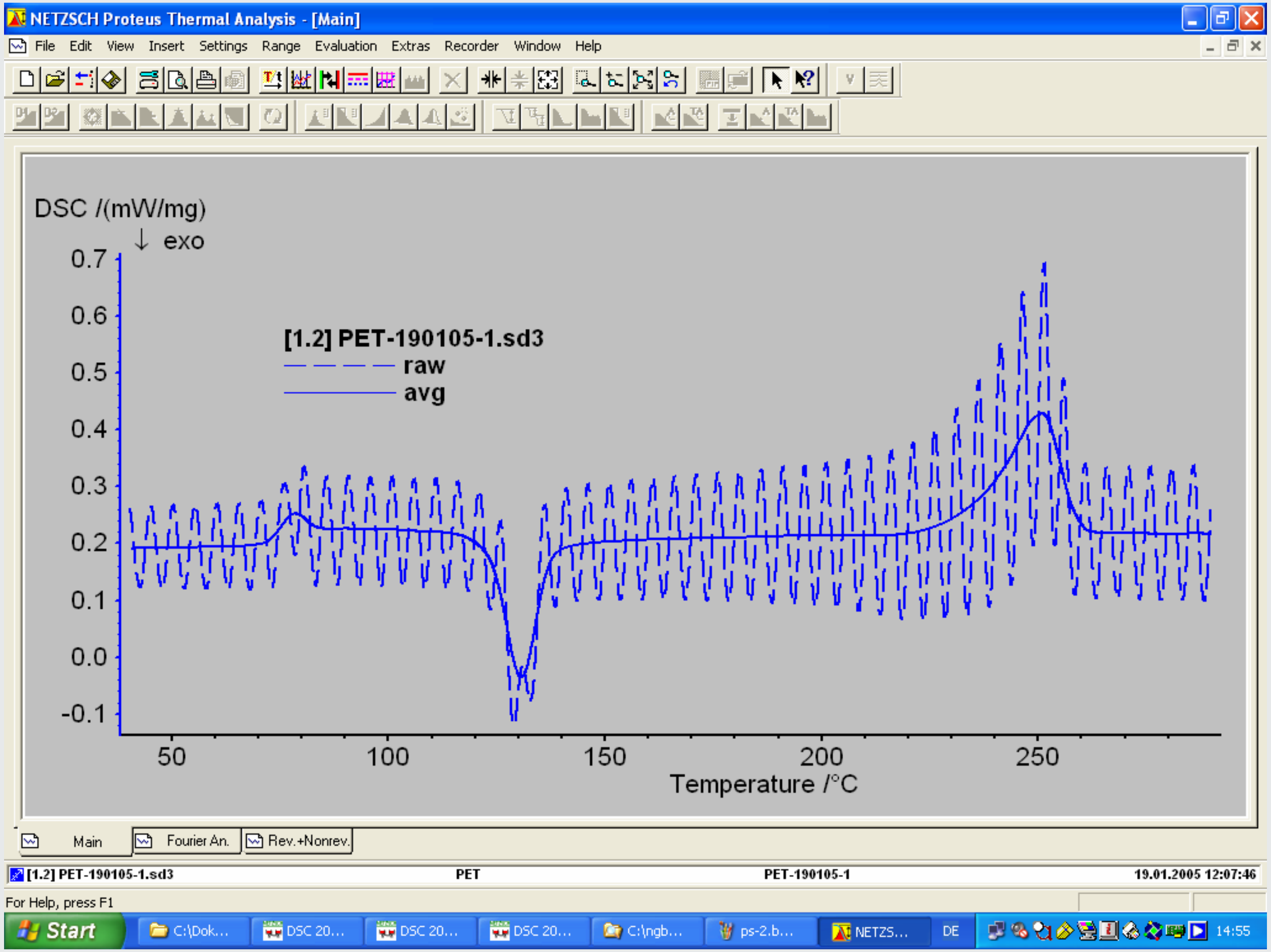
# DSC 204 F1 – TM-DSC-Parameter Input



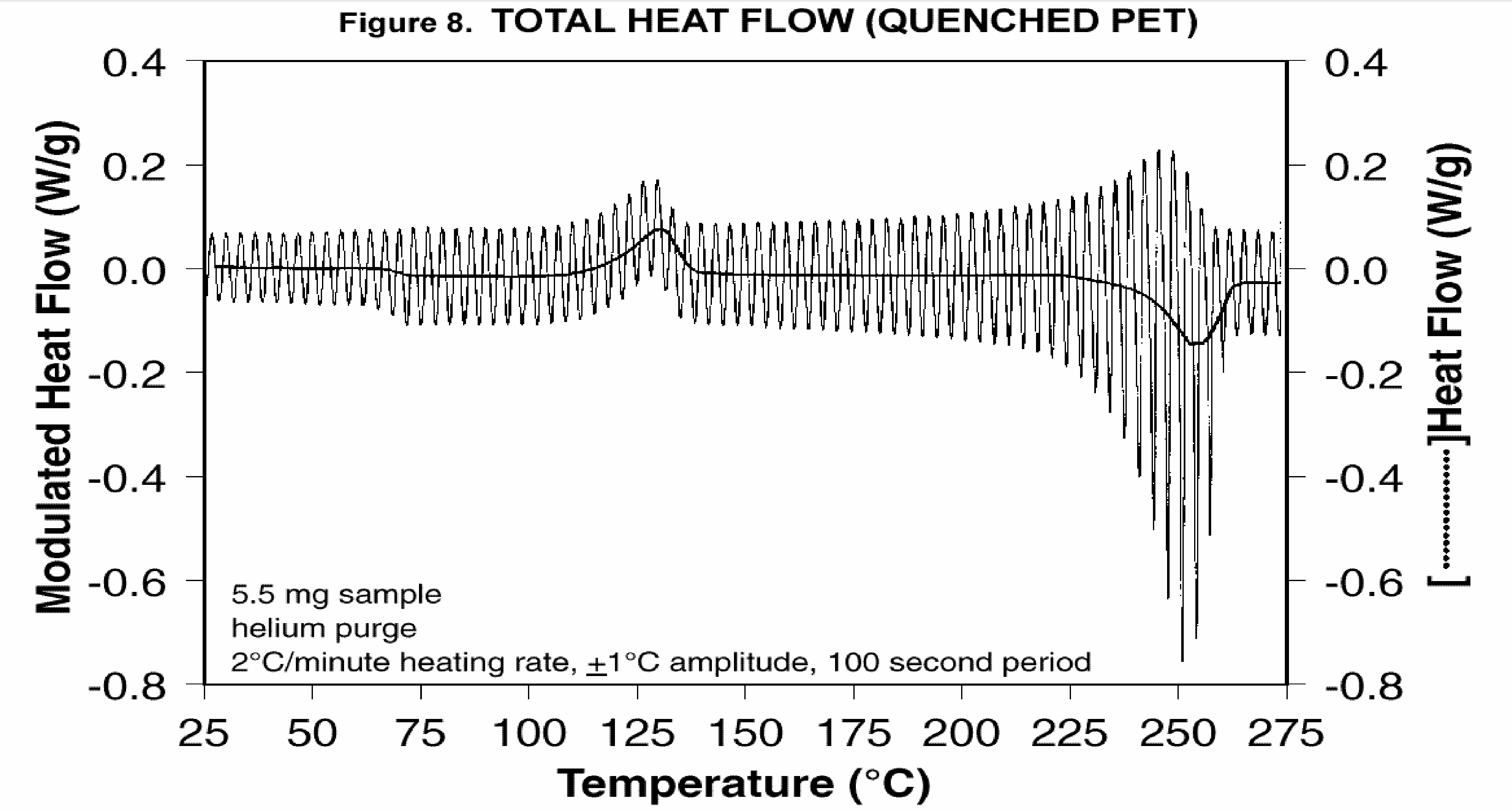
# TM-DSC - 調制熱流及調制加熱速率



# DSC 204 F1 – TM-DSC - Result

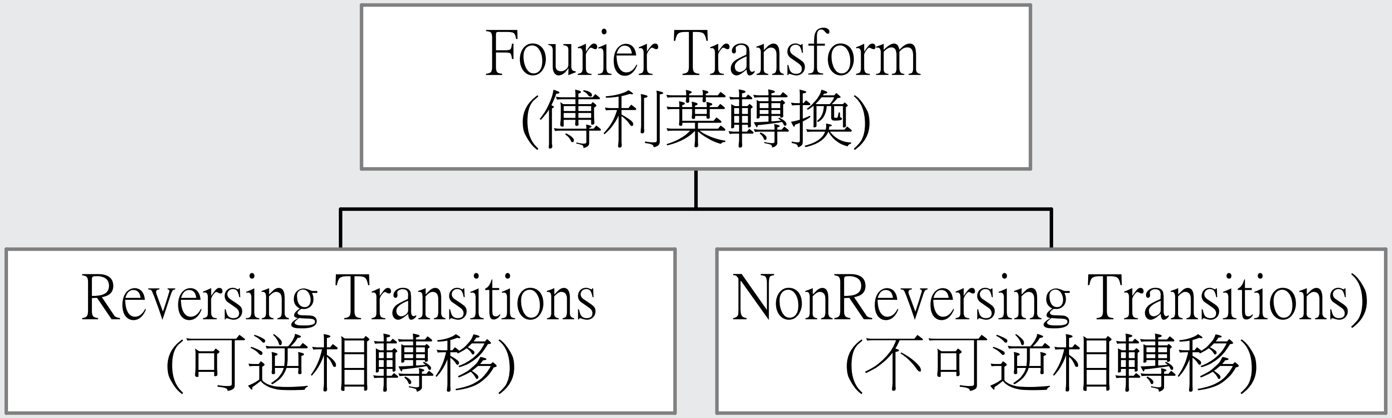


# DSC 204 F1 – TM-DSC - PET

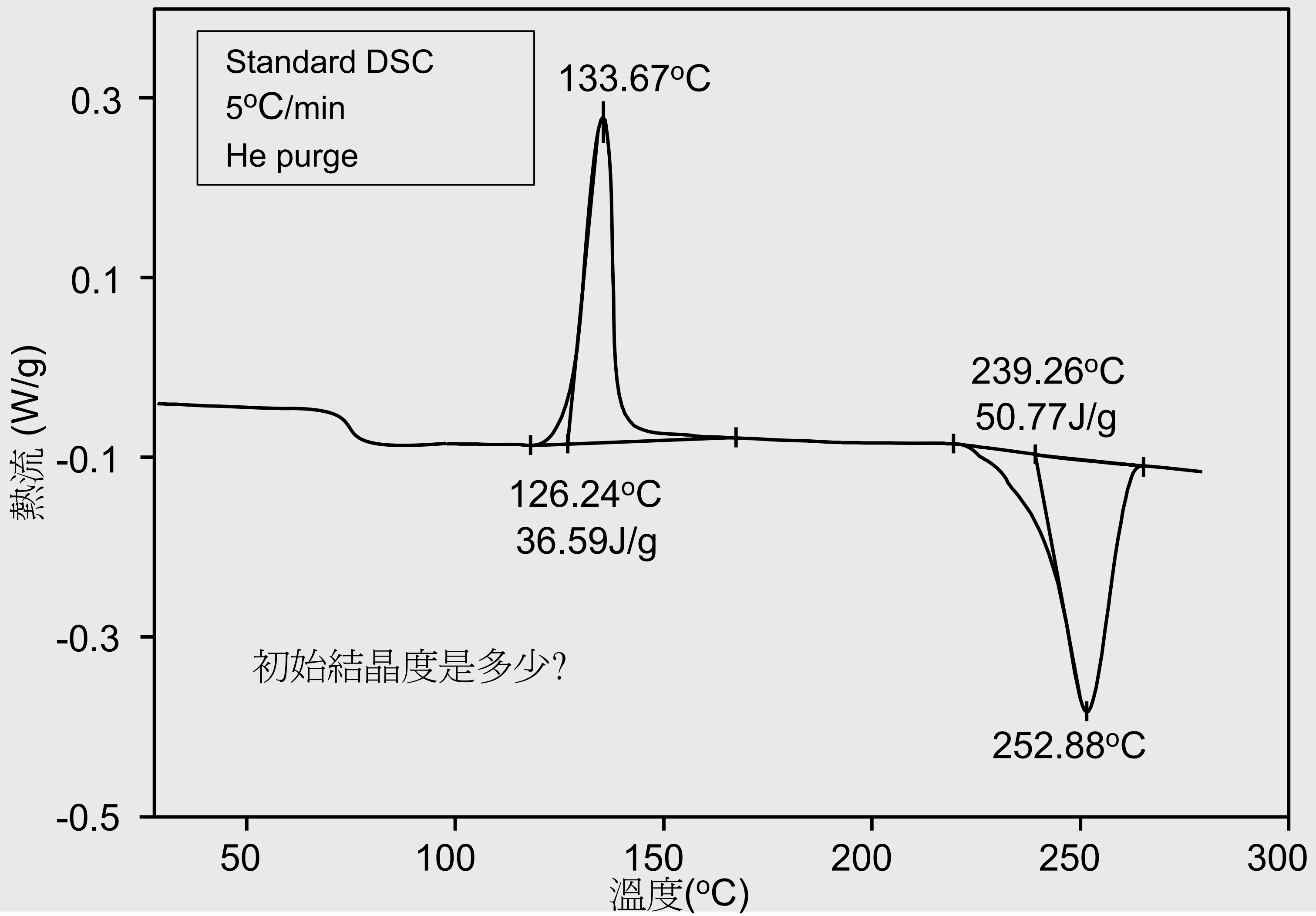


# 調制式熱流轉換原理

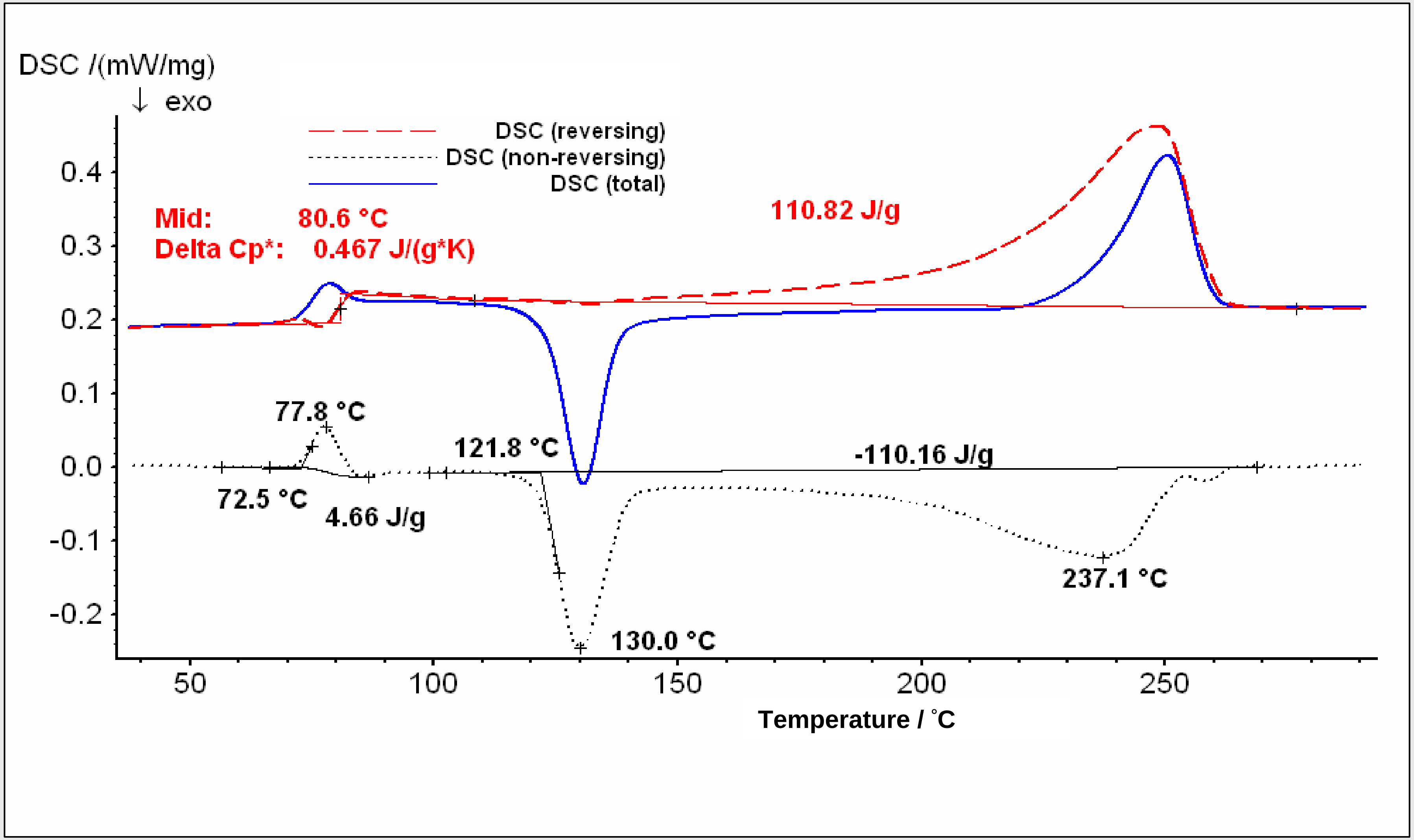
## Temperature Modulated Heat Flow
## (調制式熱流)



# 驟冷的PET － 初始結晶度用傳統DSC量測

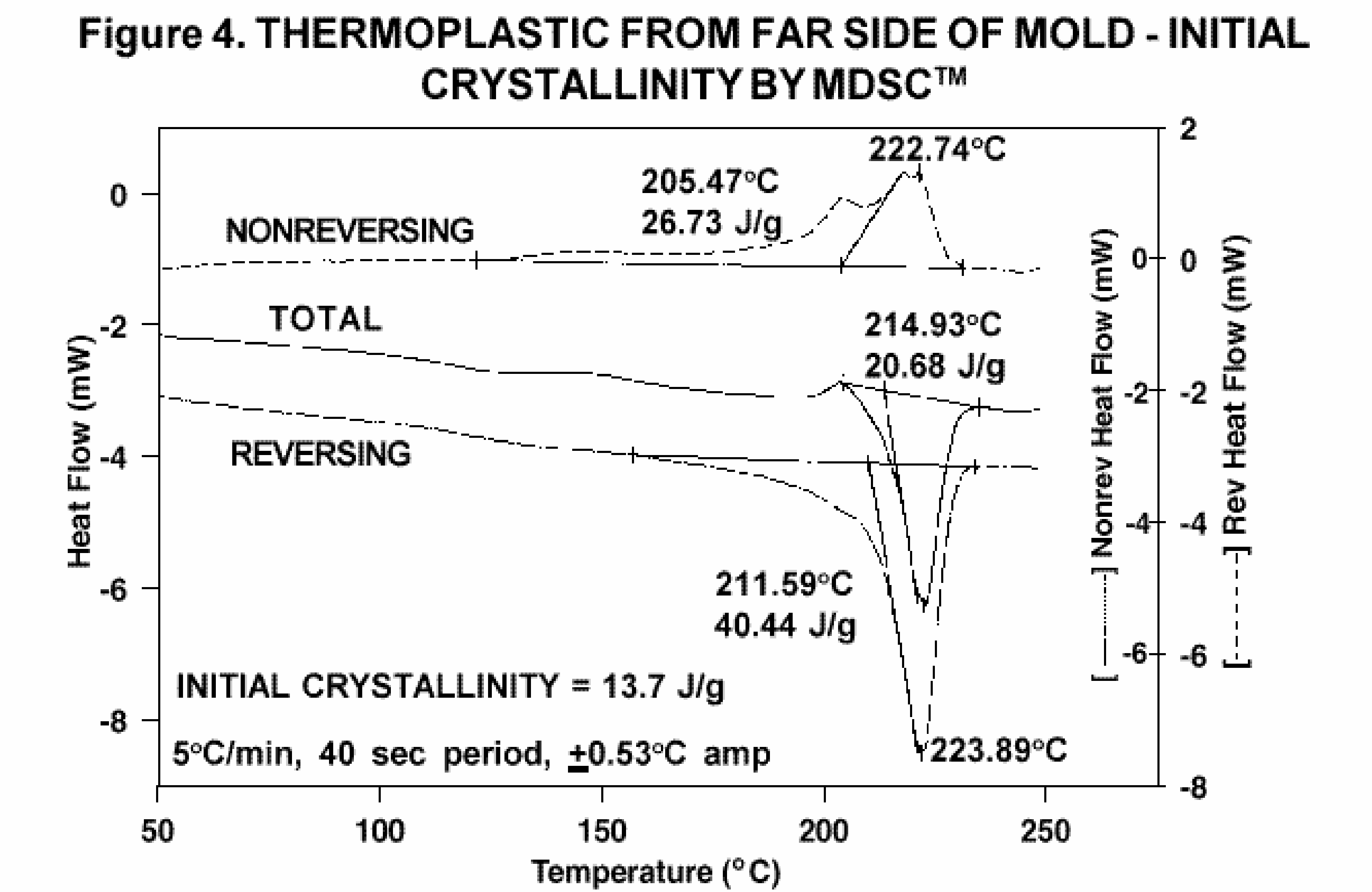


# DSC 204 F1 – TM-DSC - PET

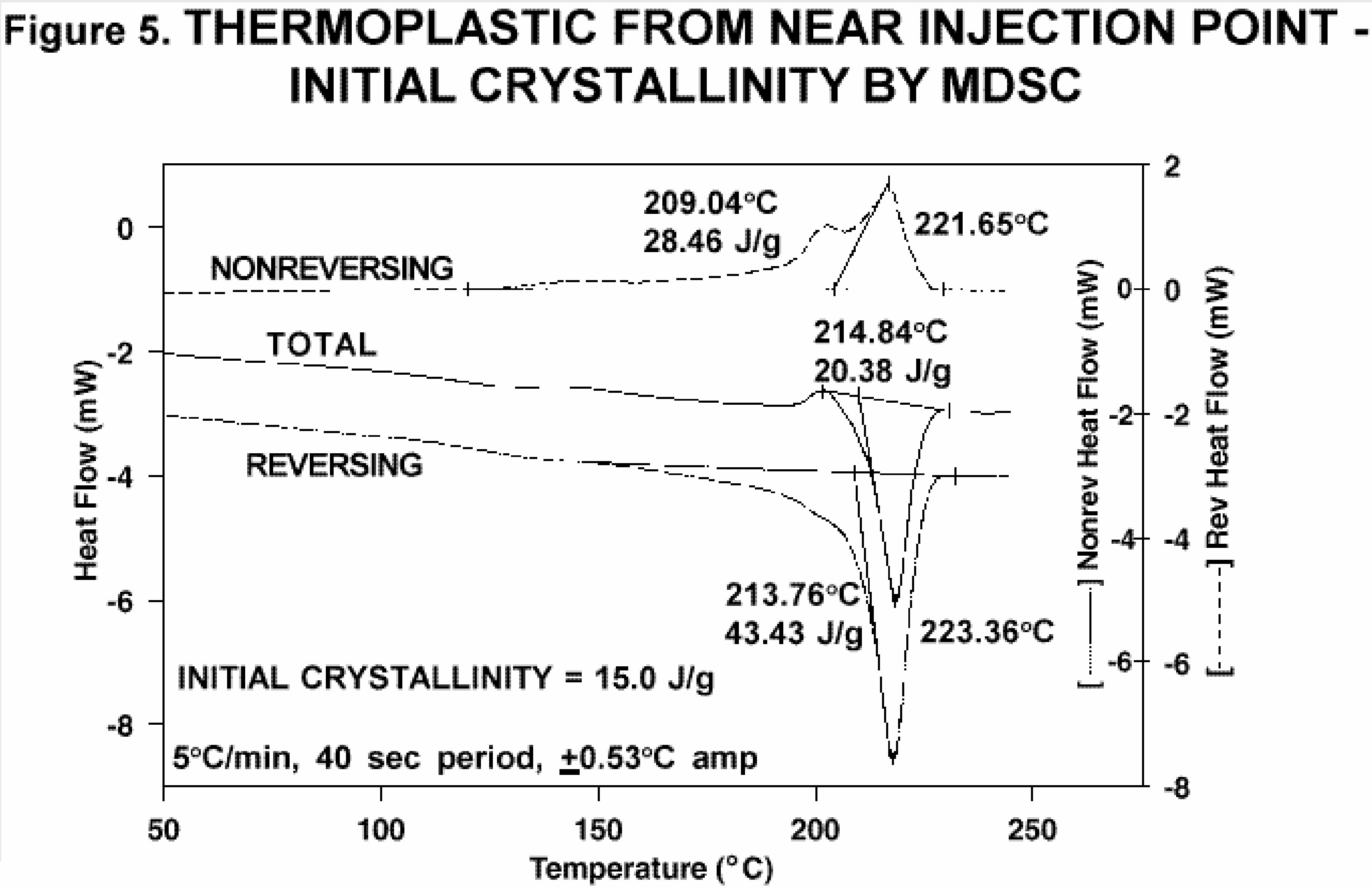


# 成型後距注射點較遠處-TM-DSC量測初始結晶度

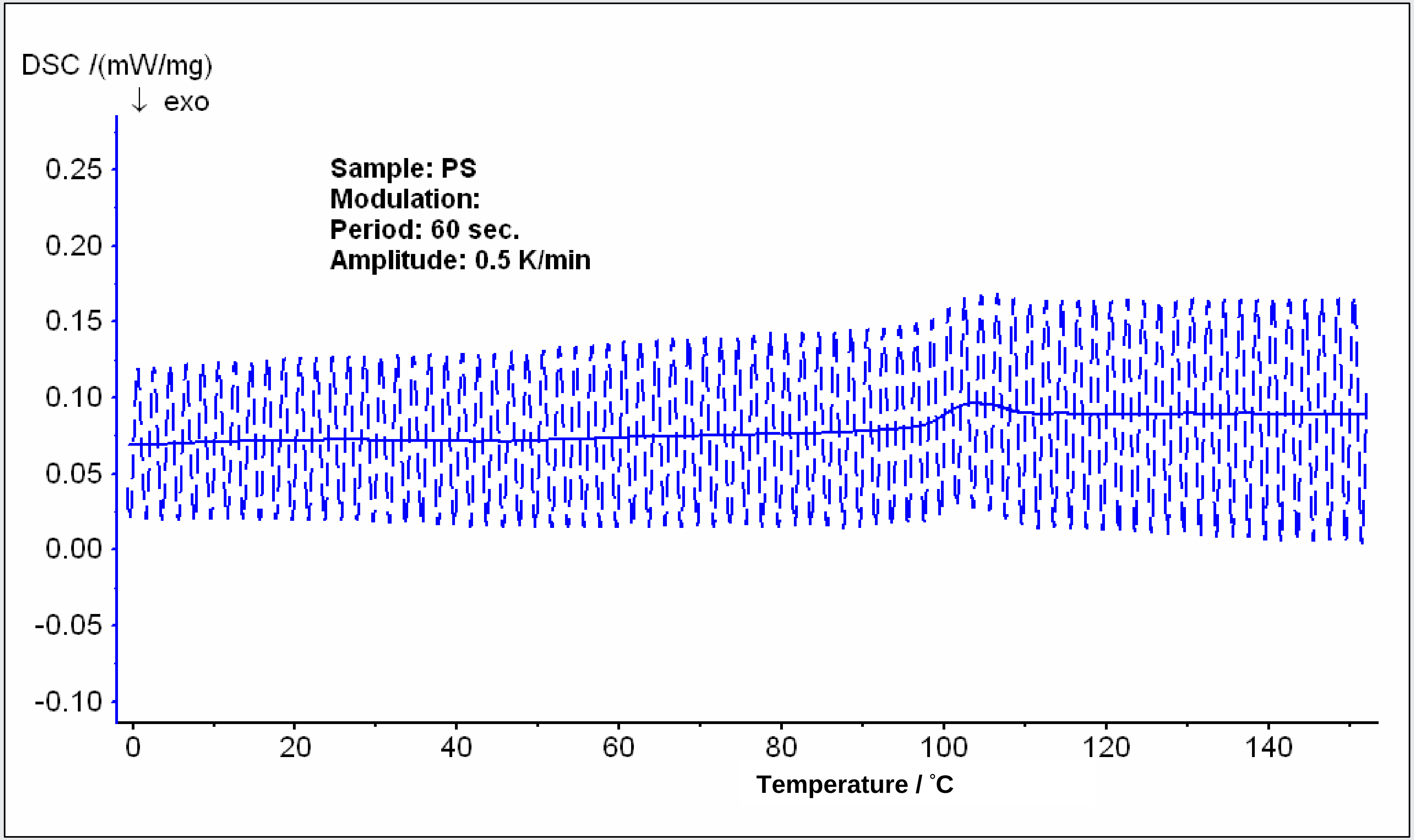
NETZSCH



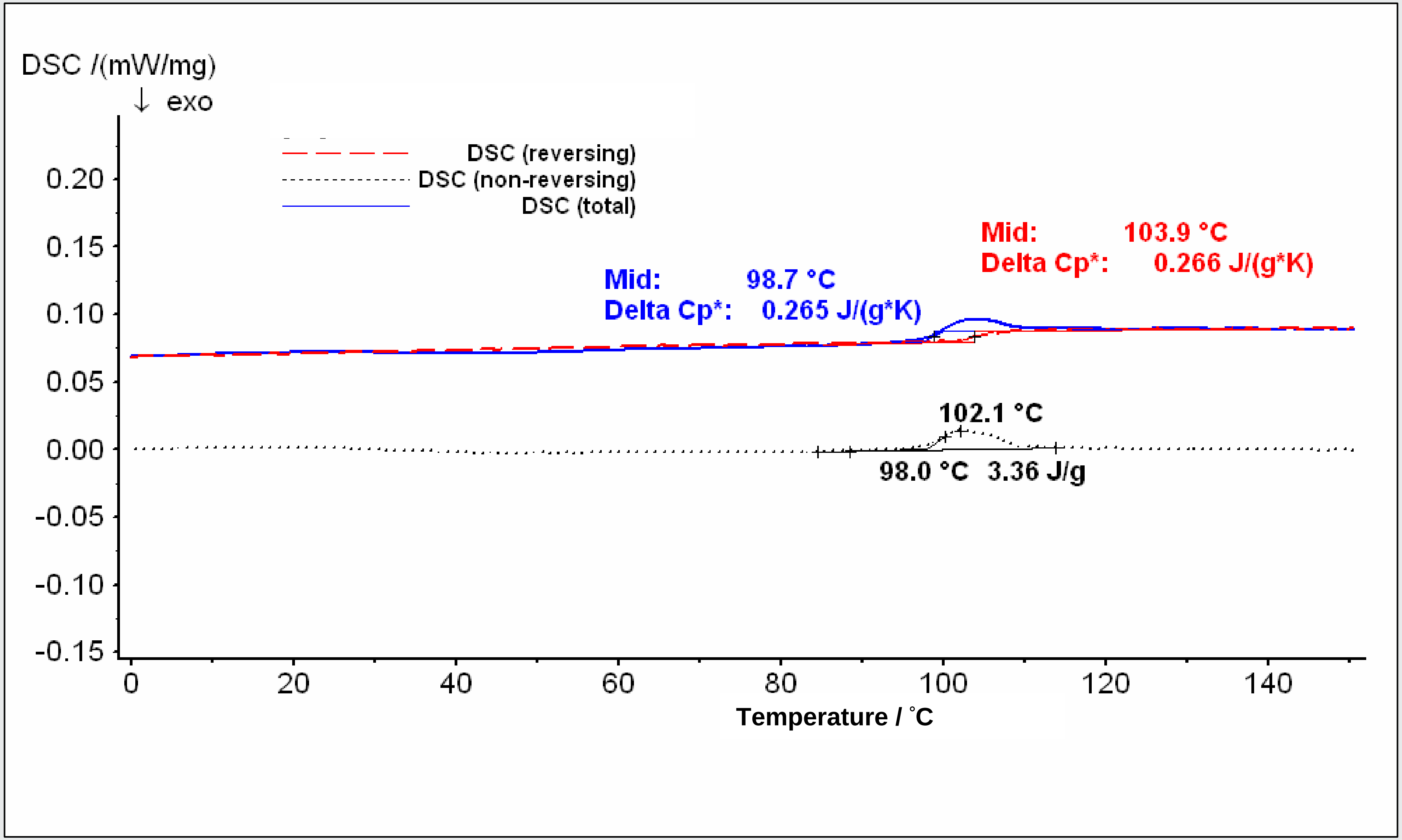
# 成型後接近注射點處-TM-DSC量測初始結晶度



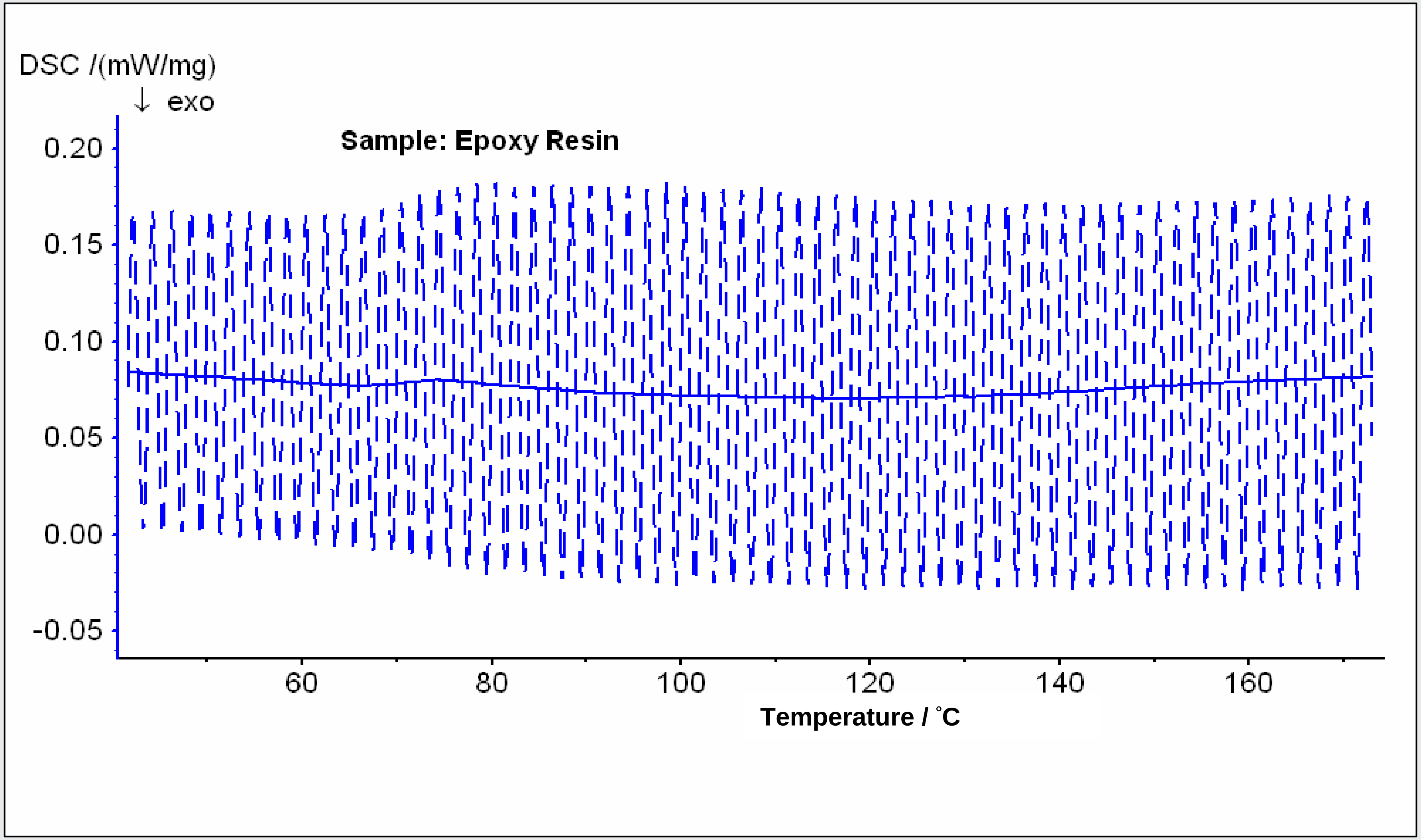
# DSC 204 F1 – TM-DSC - PS



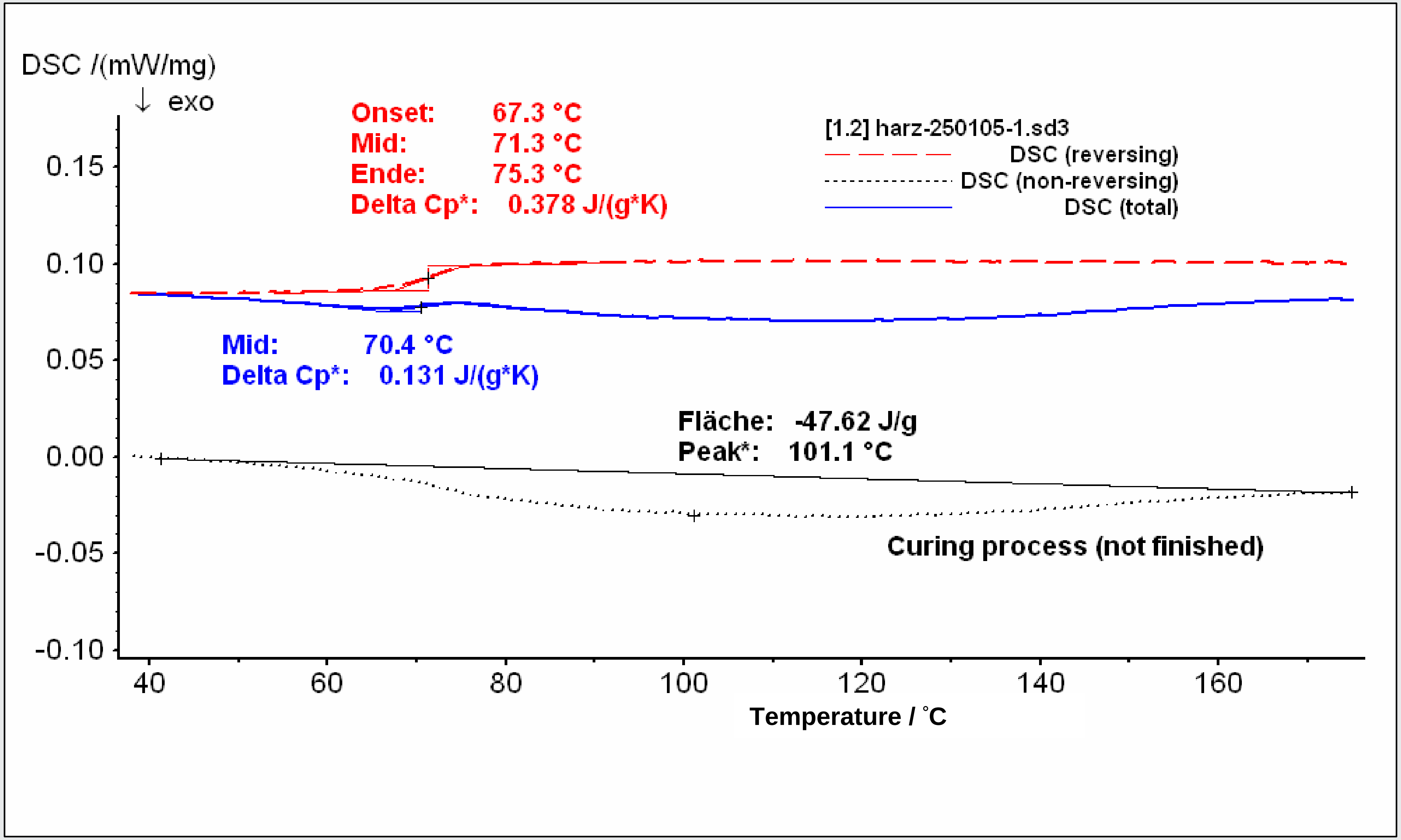
# DSC 204 F1 – TM-DSC - PS



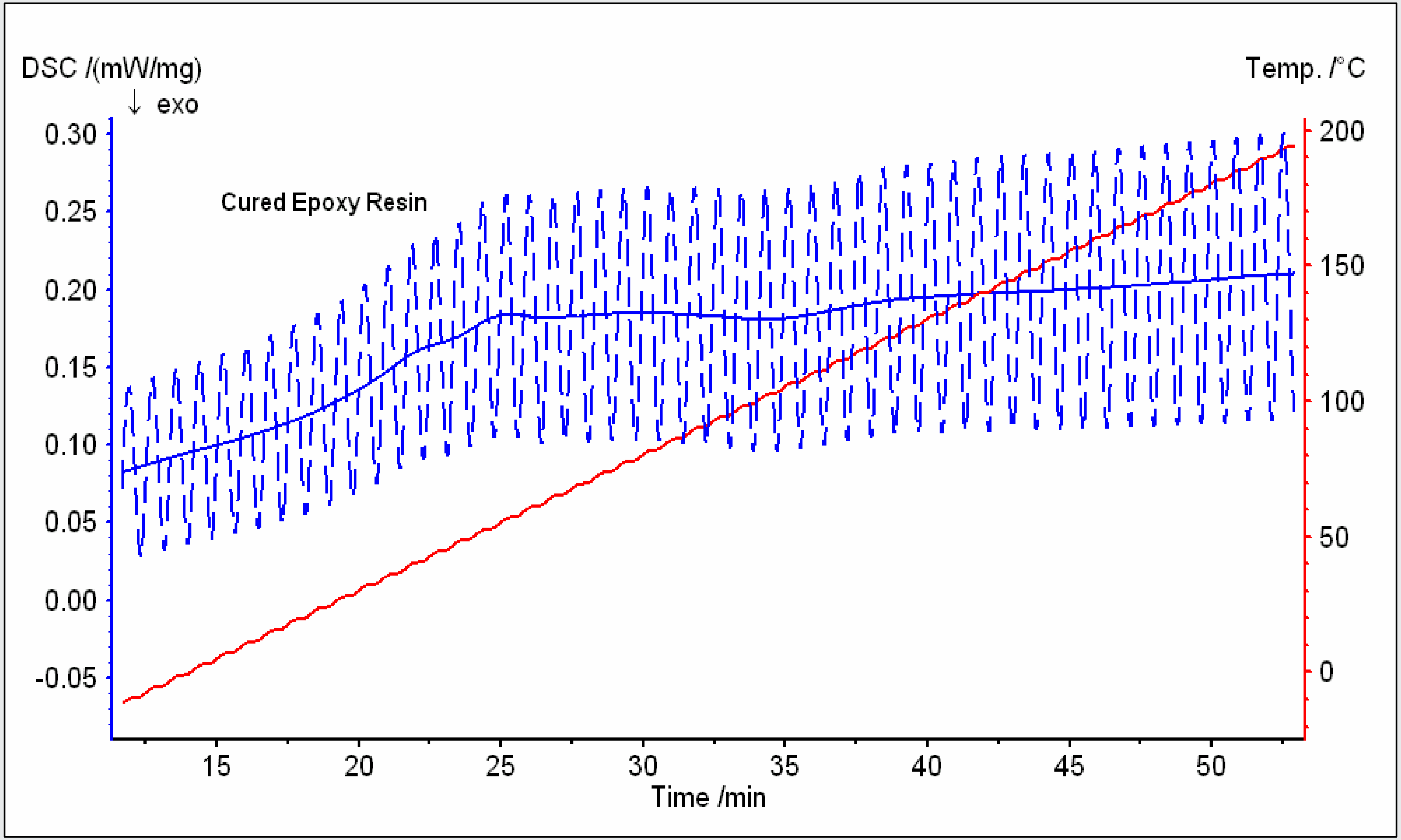
# DSC 204 F1 – TM-DSC – Curing of a Resin



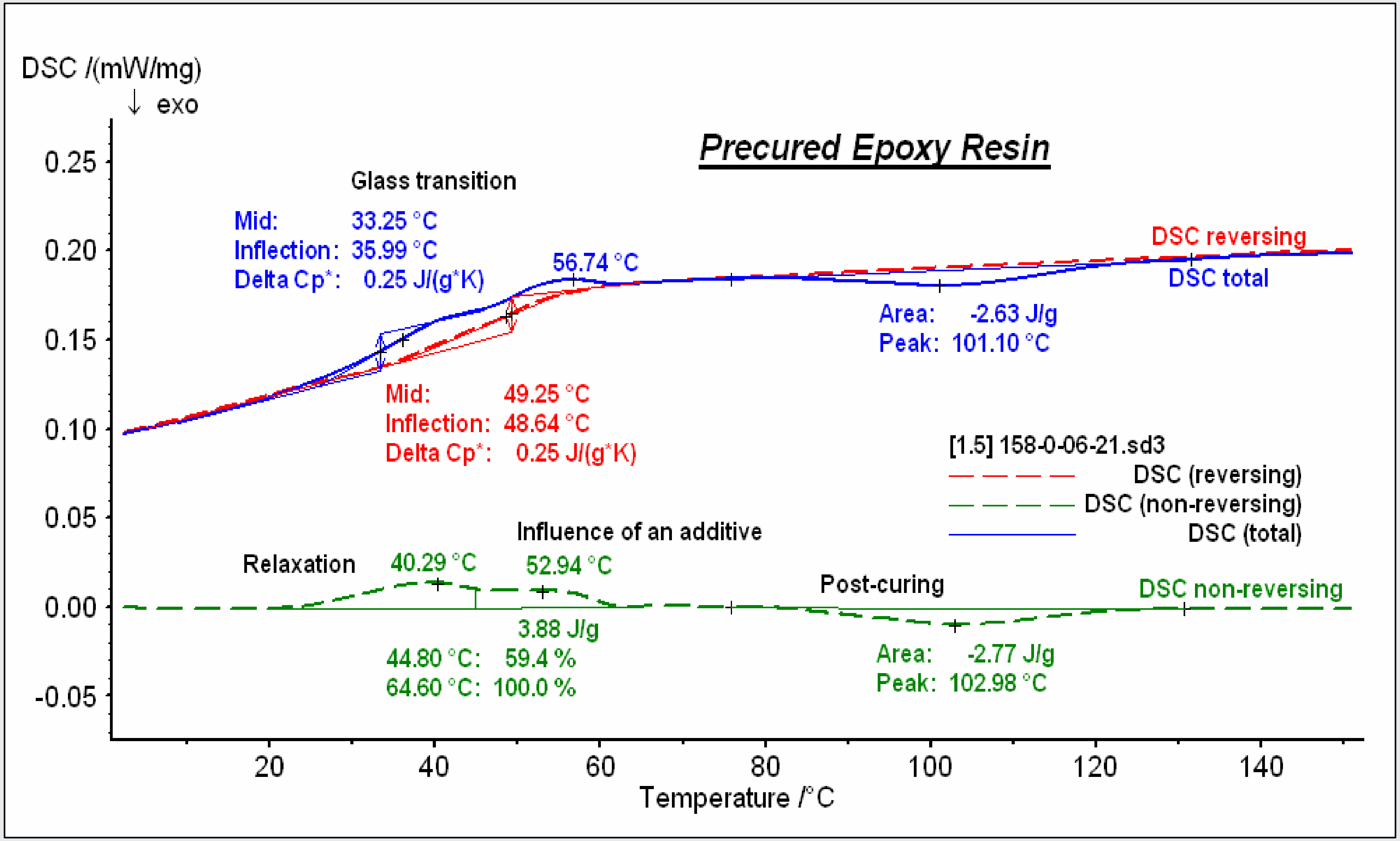
# DSC 204 F1 – TM-DSC – Curing of a Resin



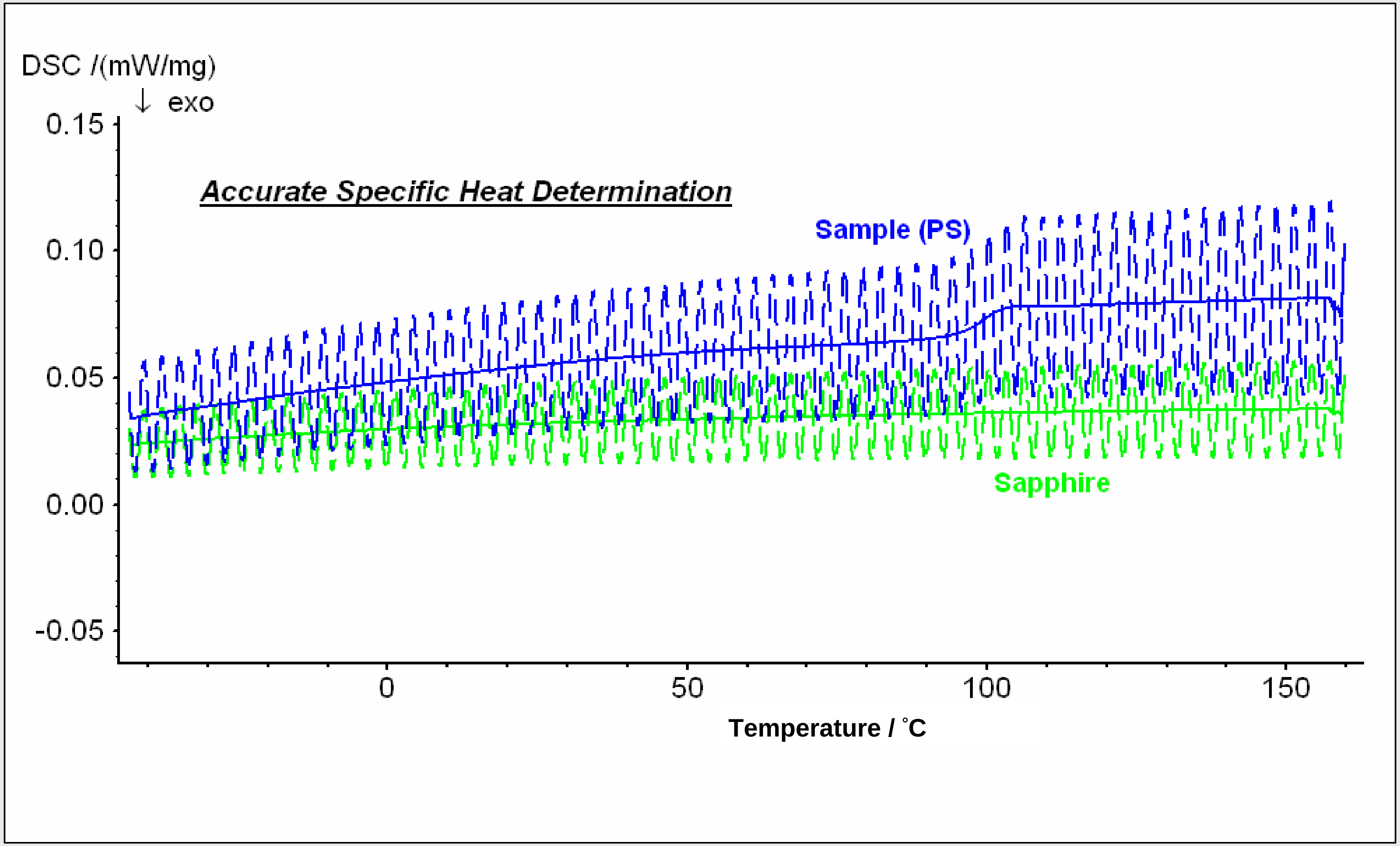
# DSC 204 F1 – TM-DSC – Post-Curing of an Epoxy Resin



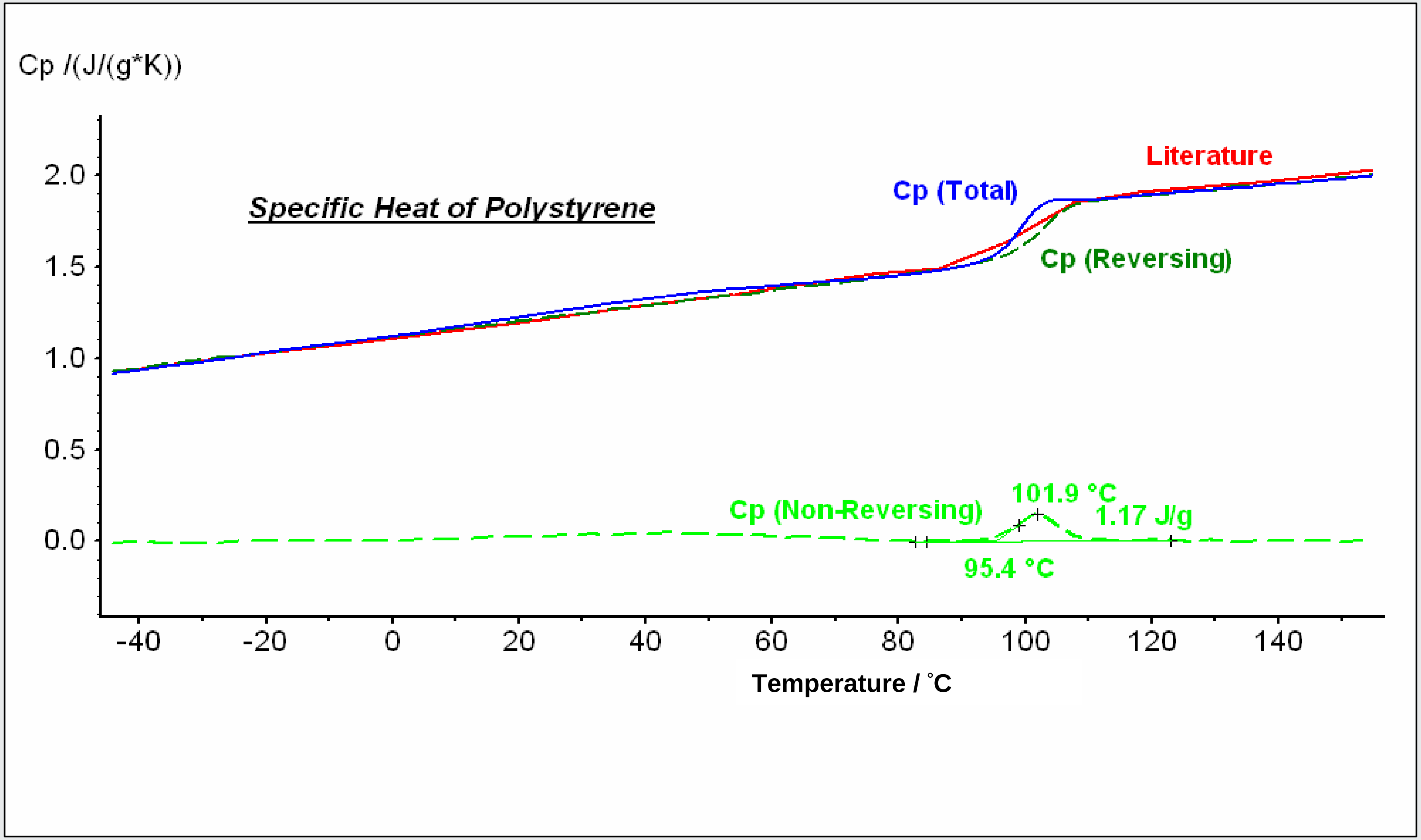
# DSC 204 F1 – TM-DSC – Post-Curing of an Epoxy Resin



# DSC 204 F1 – TMDSC – Cp-Determination



# DSC 204 F1 – TMDSC – Cp-Determination



# 傳統DSC比熱測定方法

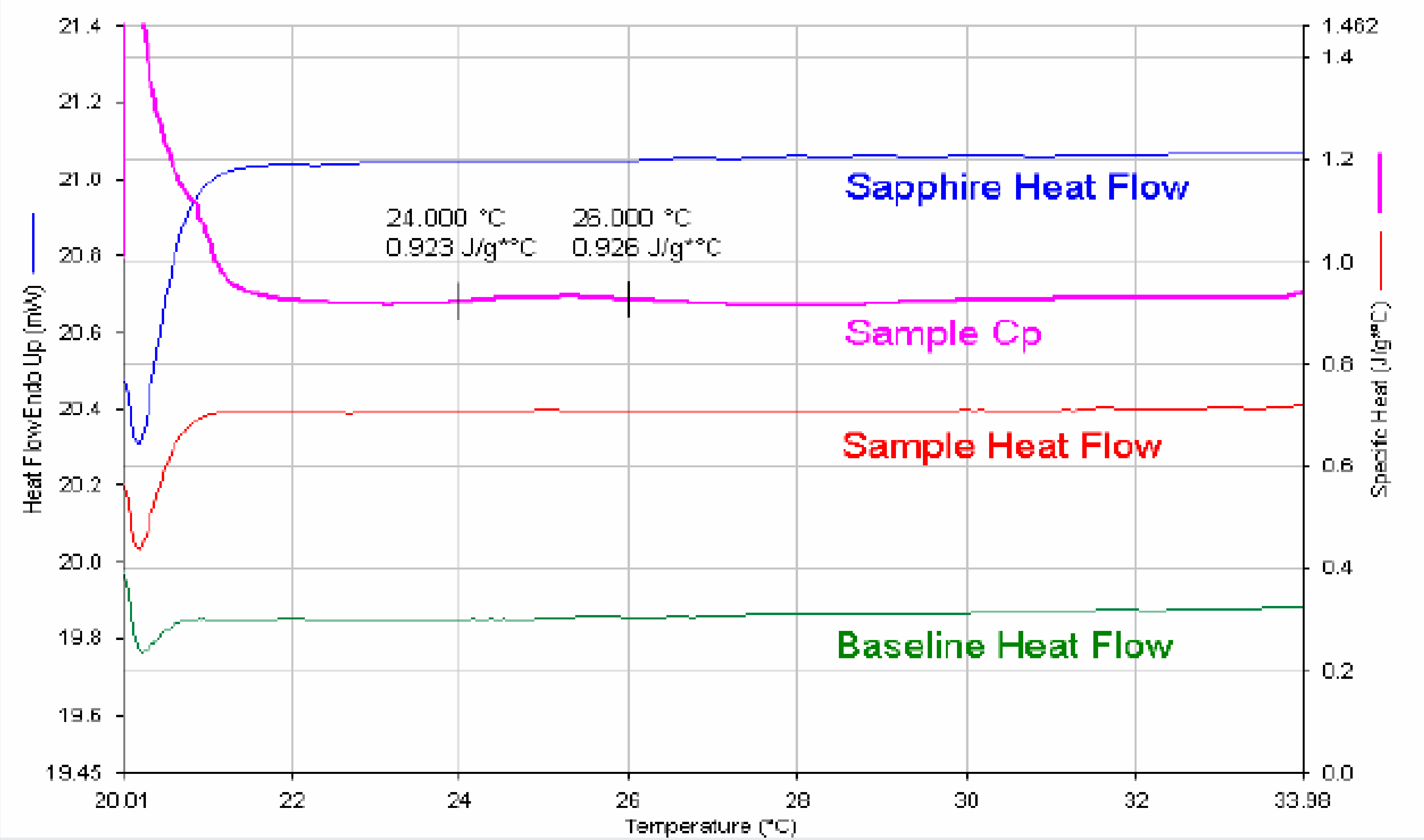
$$Cp = \frac{EQ}{RM}$$

where
Cp = Specific Heat Capacity
E = DSC Calibration Factor
Q = Heat Flow
R = Heating Rate
M = Sample Mass

$$Cp = K_{Cp} \times \frac{Q\,(sample) - Q\,(blank)}{R}$$

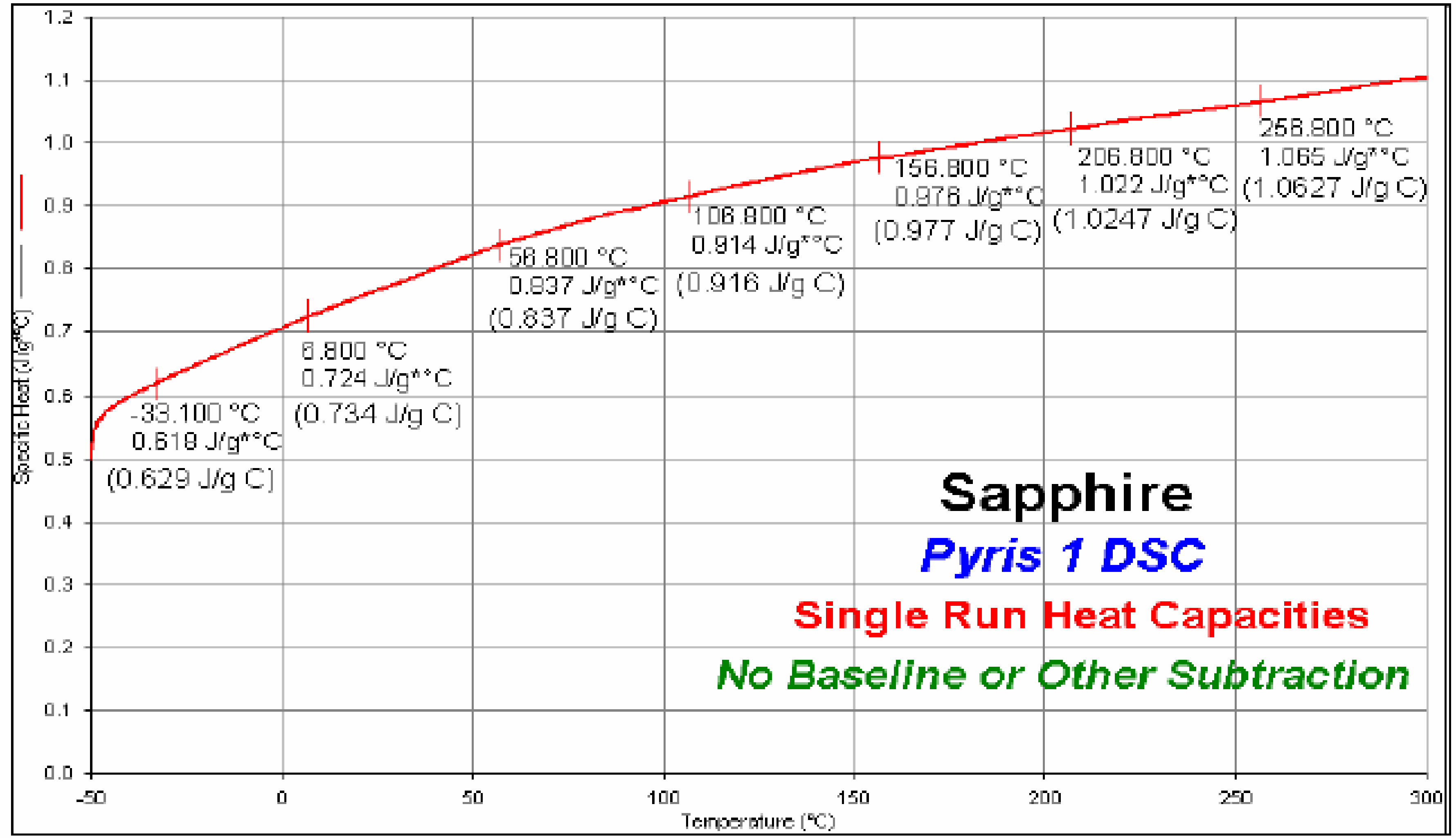
where $K_{Cp}$ = Cp cell calibration constant

# 比熱測試 -- Cp



Sapphire Heat Flow
Sample Cp
Sample Heat Flow
Baseline Heat Flow
24.000 °C
0.923 J/g*°C
26.000 °C
0.926 J/g*°C
Heat Flow Endo Up (mW)
Specific Heat (J/g*°C)
Temperature (°C)
21.4
21.2
21.0
20.8
20.6
20.4
20.2
20.0
19.8
19.6
19.45
1.462
1.4
1.2
1.0
0.8
0.6
0.4
0.2
0.0
20.01
22
24
26
28
30
32
33.98

# *Sapphire Cp test*



# TM-DSC 訊號 - 比熱

比熱係由振盪熱流的幅度除以振盪加熱速率的幅度而得.

定義 : 讓物質每增加1°C所需要的熱能.

計算 : 利用MDSC獲得比熱的基礎,可由檢視傳統DSC在若干不同加熱速率實驗下計算比熱的經驗而得.

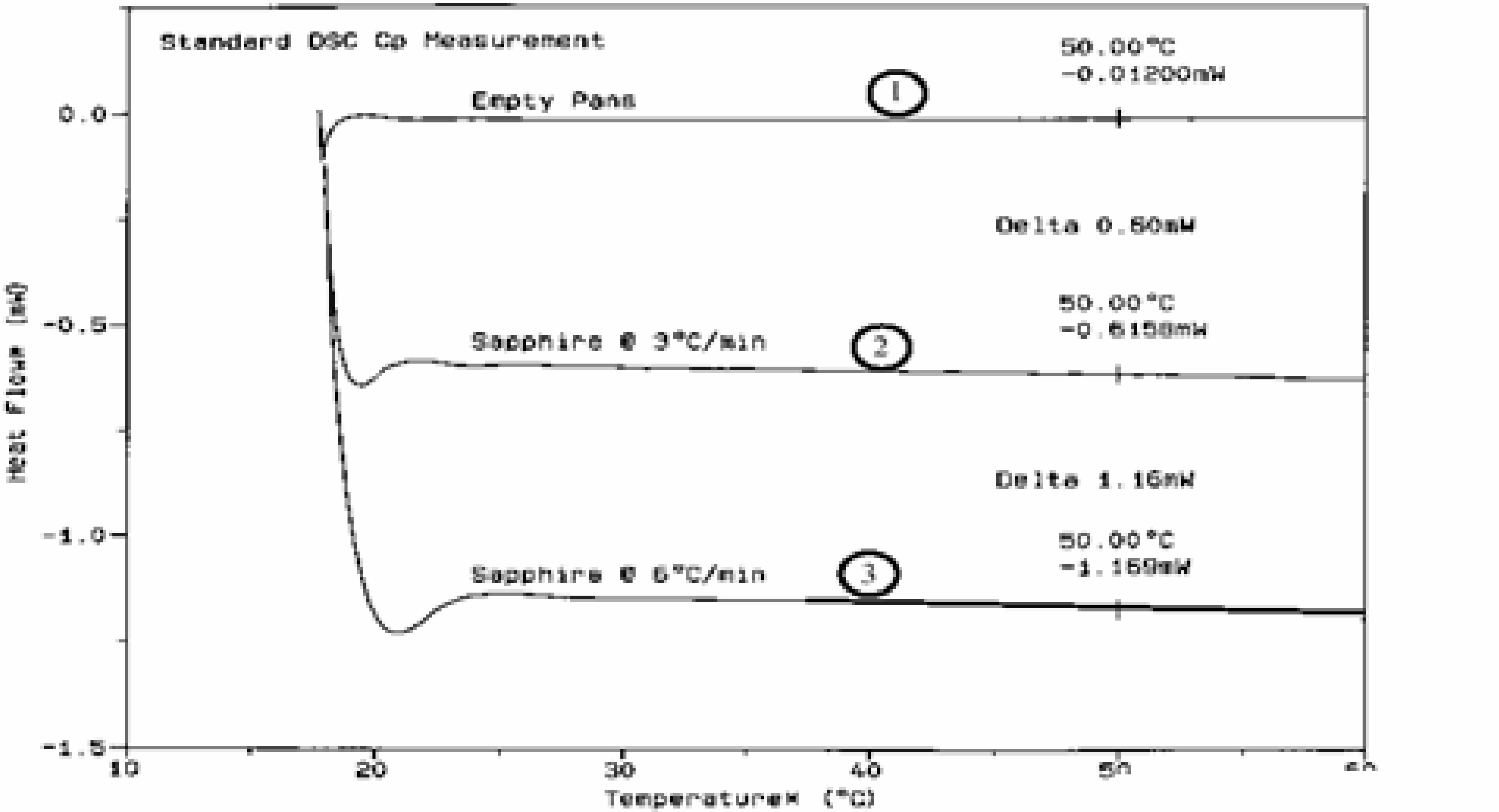
傳統DSC(在兩個不同加熱速率下)

$$Cp = KCp \times \frac{\text{第2加熱速率下的熱流值} - \text{第1加熱速率下的熱流值}}{\text{第2加熱速率} - \text{第1加熱速率}}$$

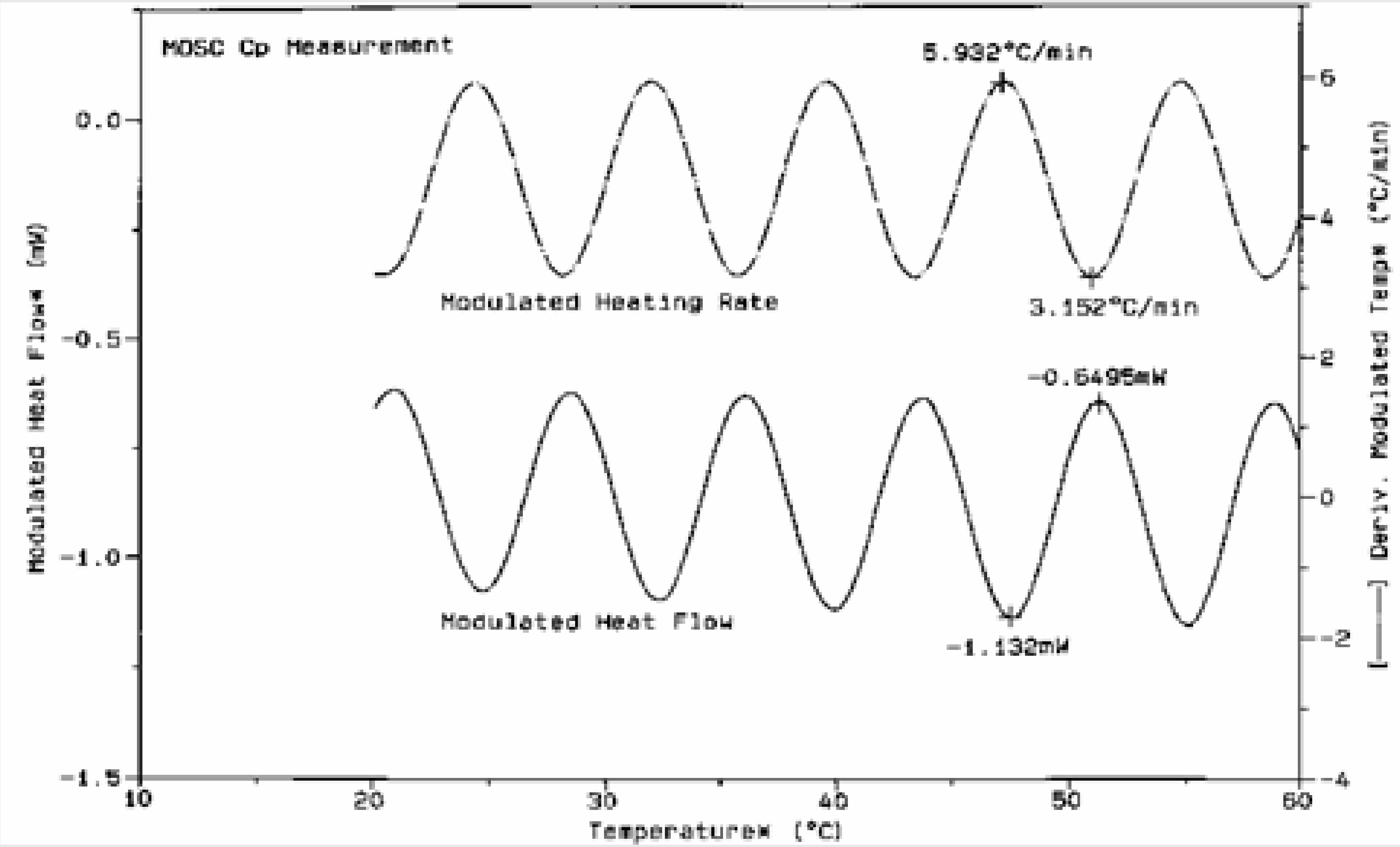
$$= mJ / {}^{\circ}C$$

where : KCp = 校正常數

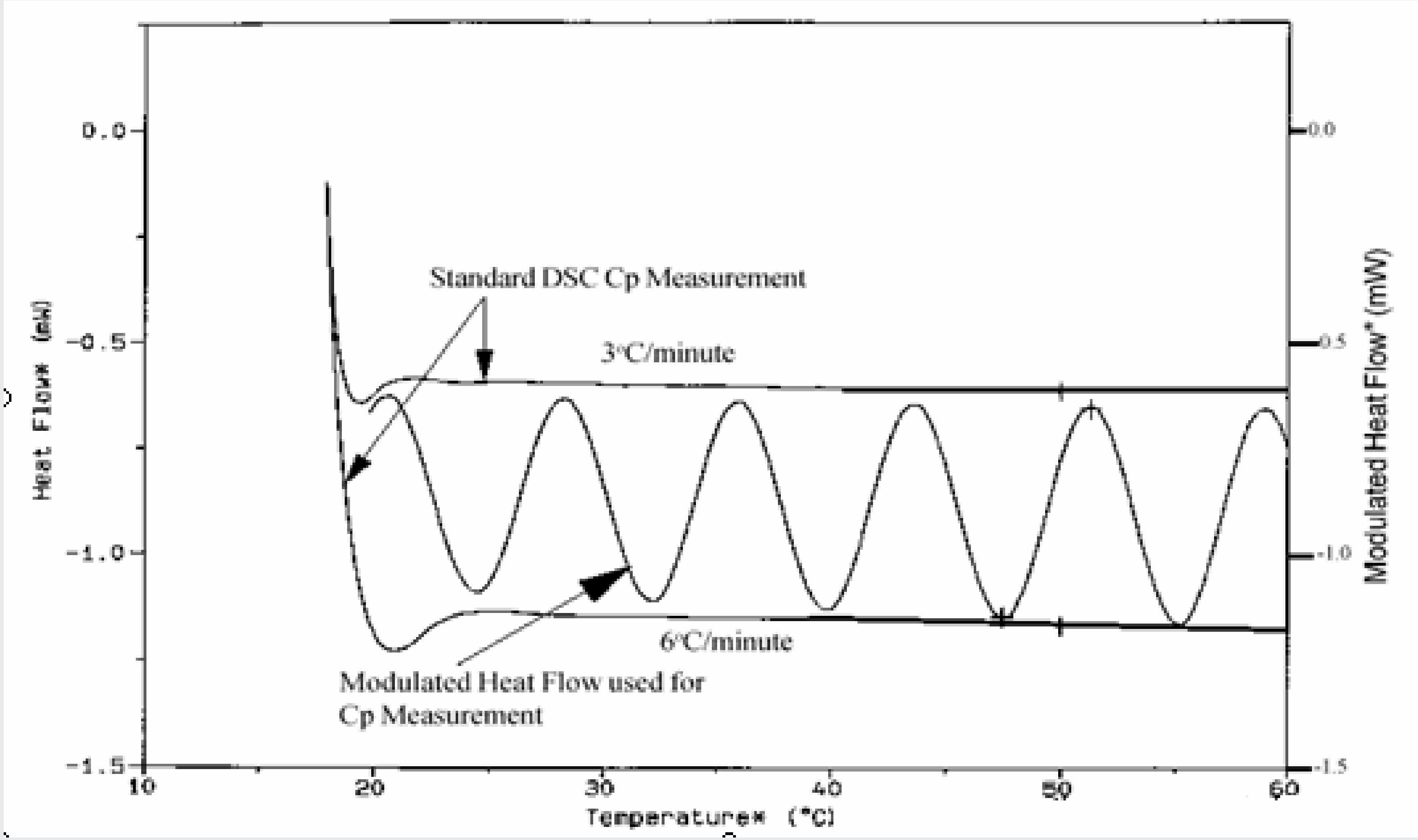
# 傳統 DSC 比熱測量圖譜



# 調制式 TM-DSC 比熱測量圖譜



# TM-DSC vs. DSC 比熱測量比較

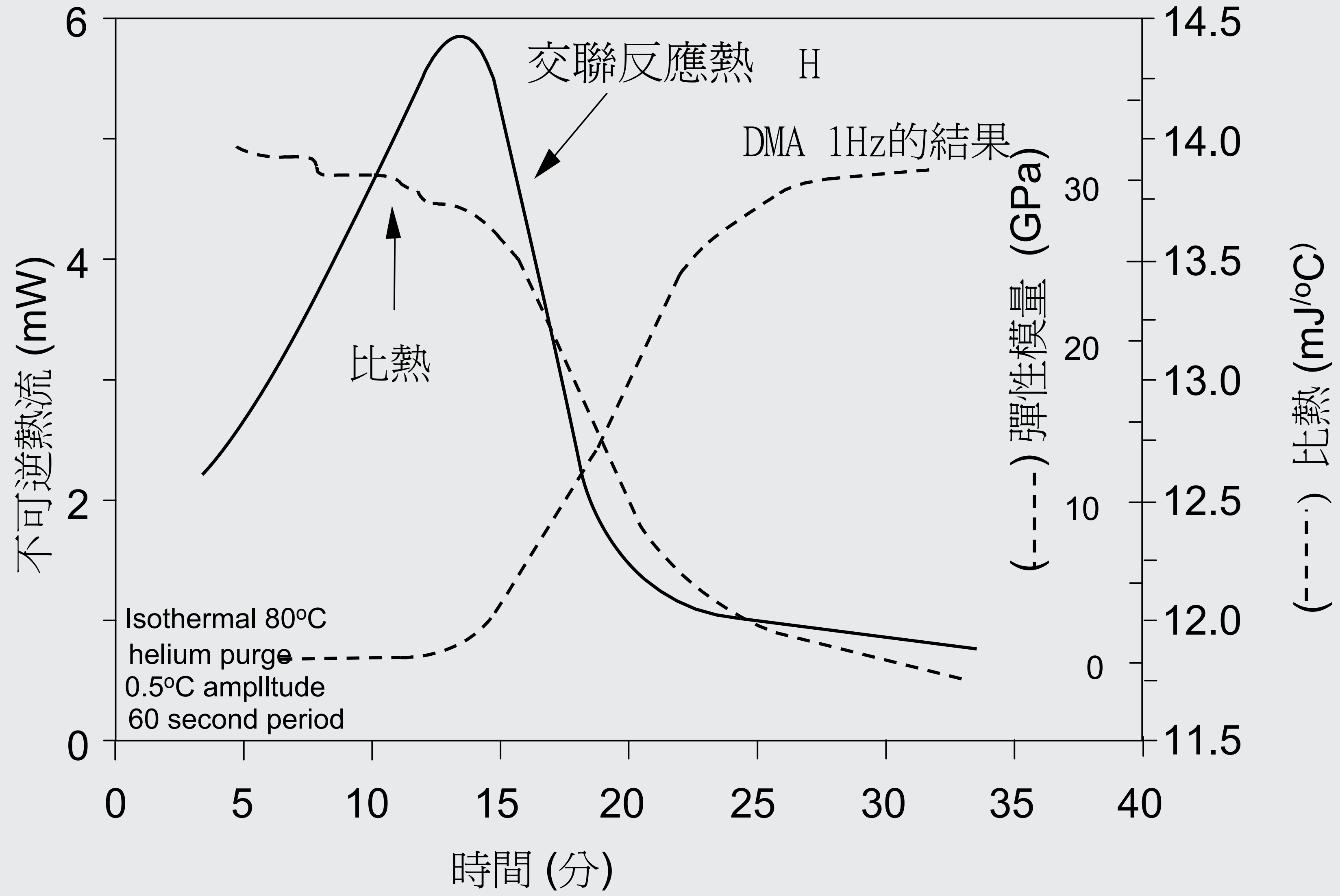


# TM-DSC 測出數據與文獻的比較:

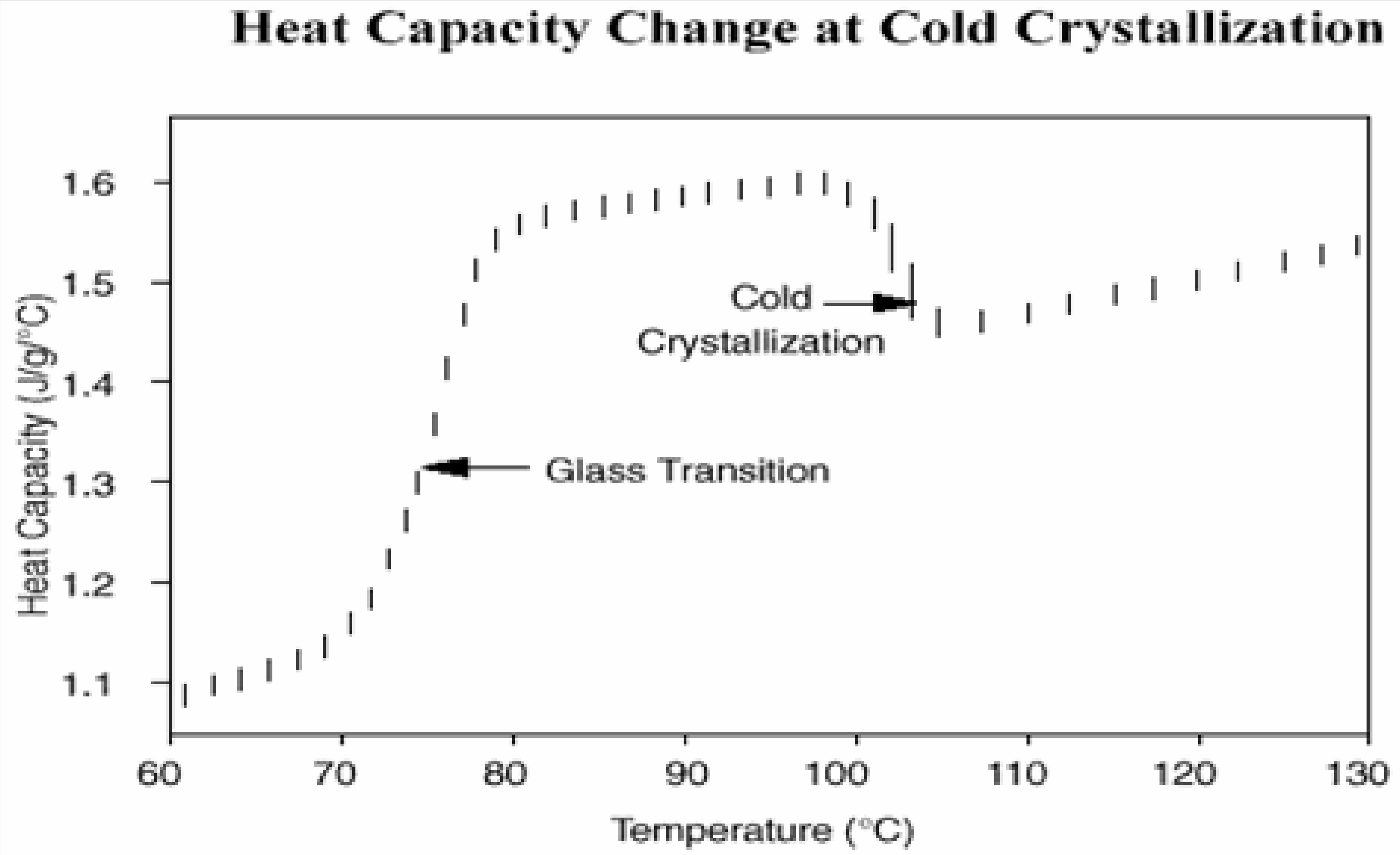
## Copper Heat Capacity

| Temp. (°C) | Cp (MDSC) (J/g/°C) | Cp (Liter.) (J/g/°C) |
|---|---|---|
| -154.3 | 0.2462 | 0.2812 |
| -102.4 | 0.3116 | 0.3401 |
| -51.5 | 0.3432 | 0.3656 |
| -1.1 | 0.3639 | 0.3783 |
| 49.2 | 0.3737 | 0.3877 |
| 99.3 | 0.3784 | 0.3971 |
| 199.4 | 0.3863 | 0.4128 |
| 249.4 | 0.3977 | 0.4187 |
| 299.5 | 0.4175 | 0.4284 |

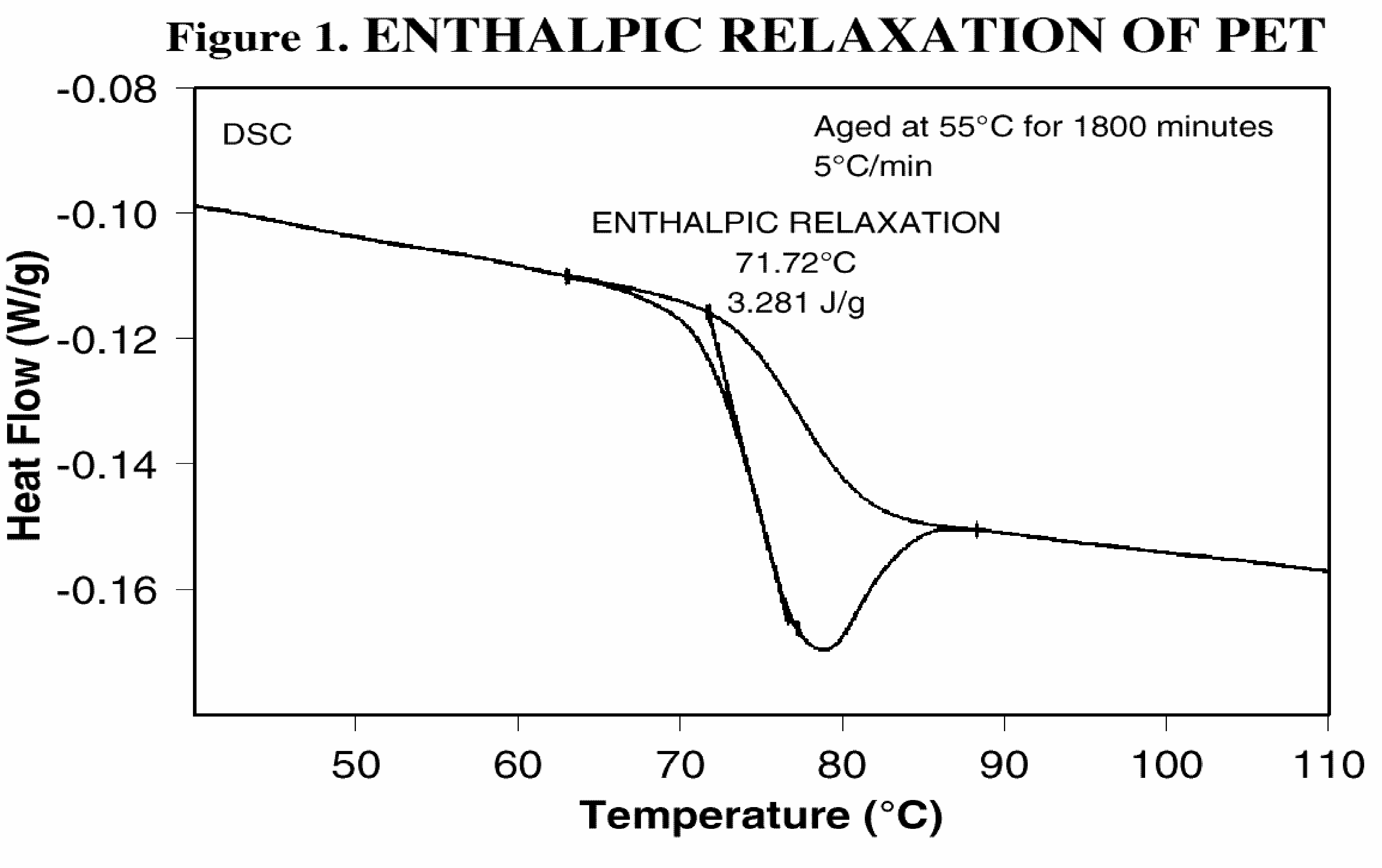
# 環氧樹脂在擬恆溫下的交聯



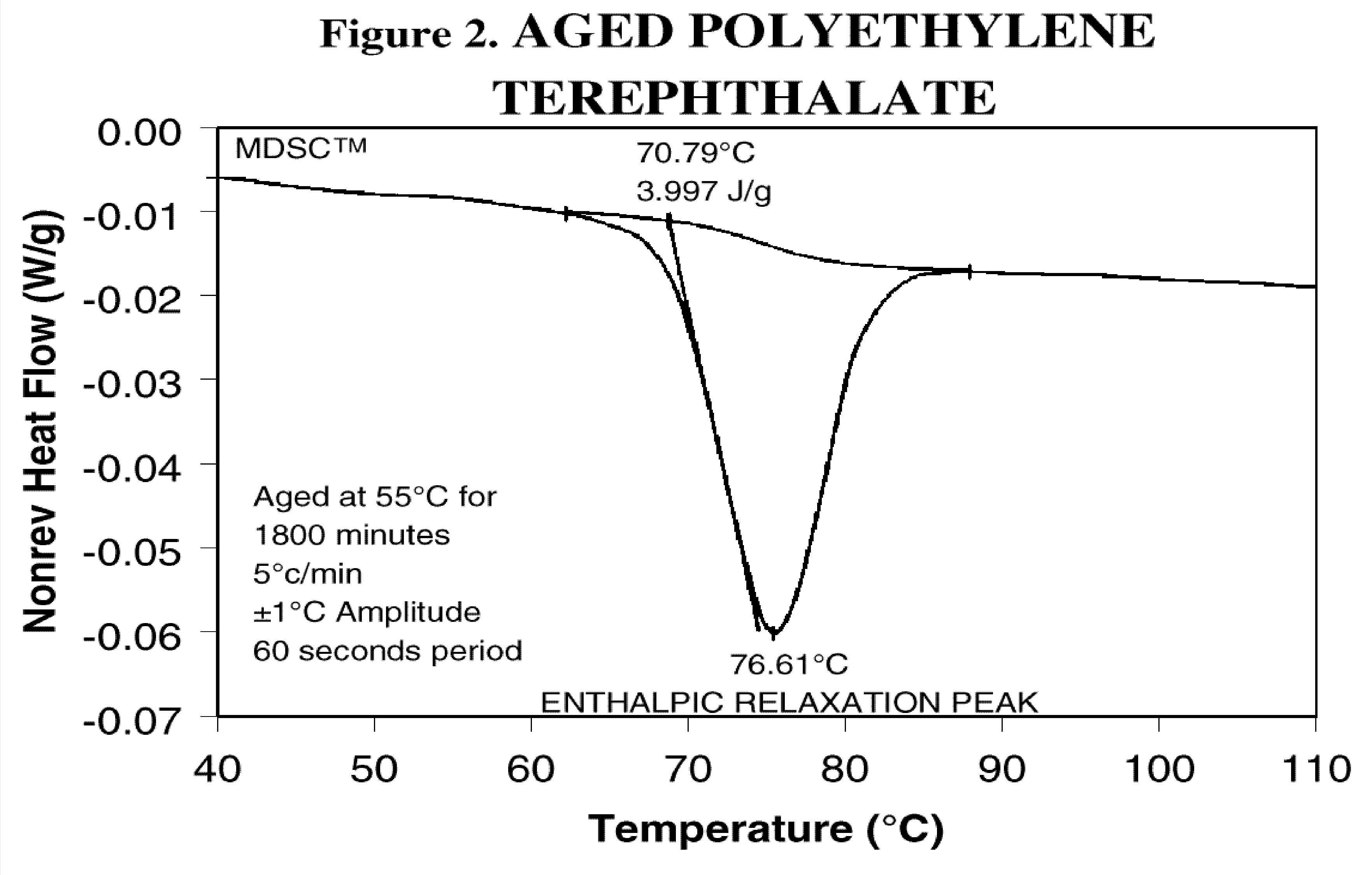
# TM-DSC 在擬恆溫下冷結晶測量:



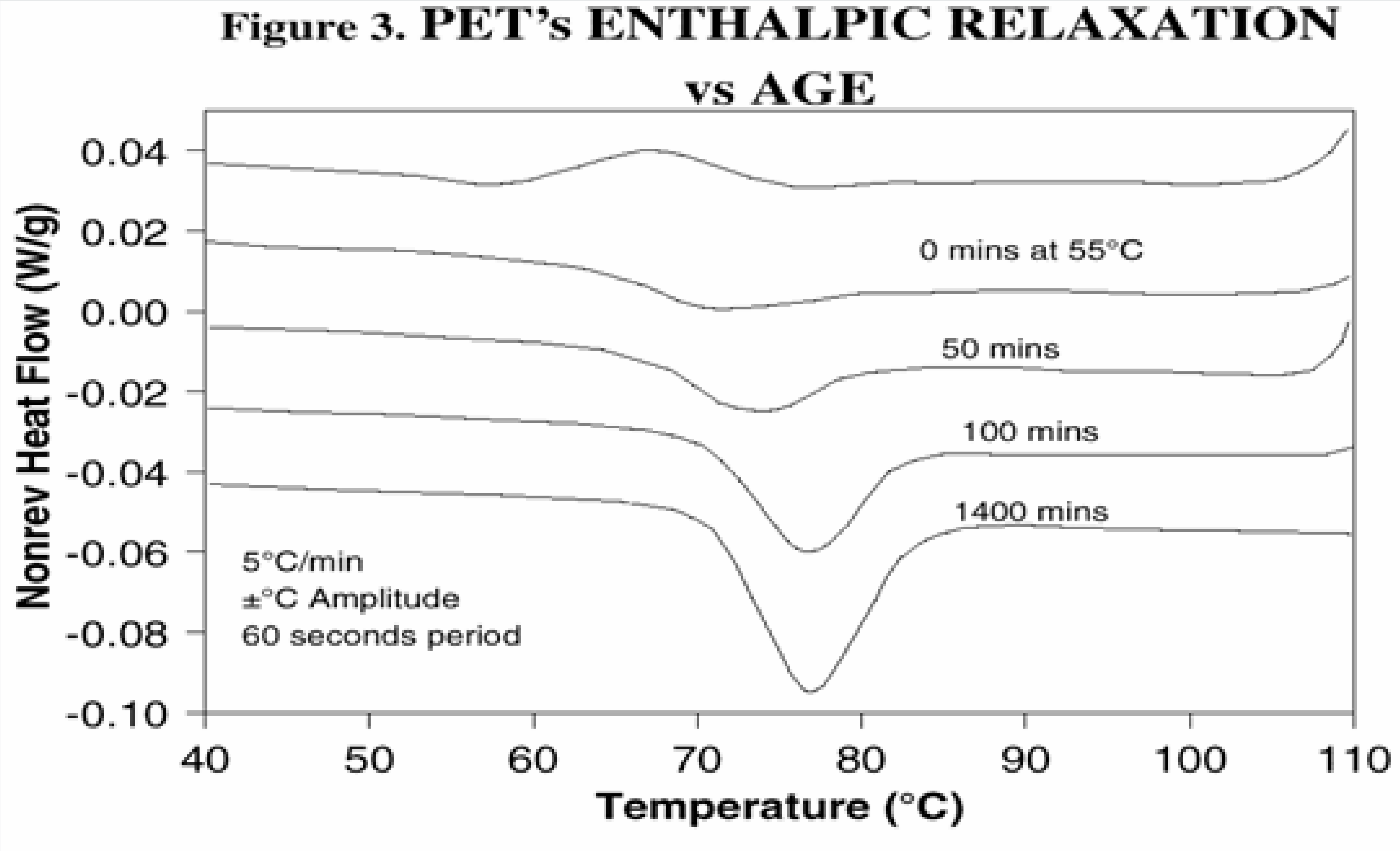
# PET焓鬆馳 - 傳統DSC之圖譜



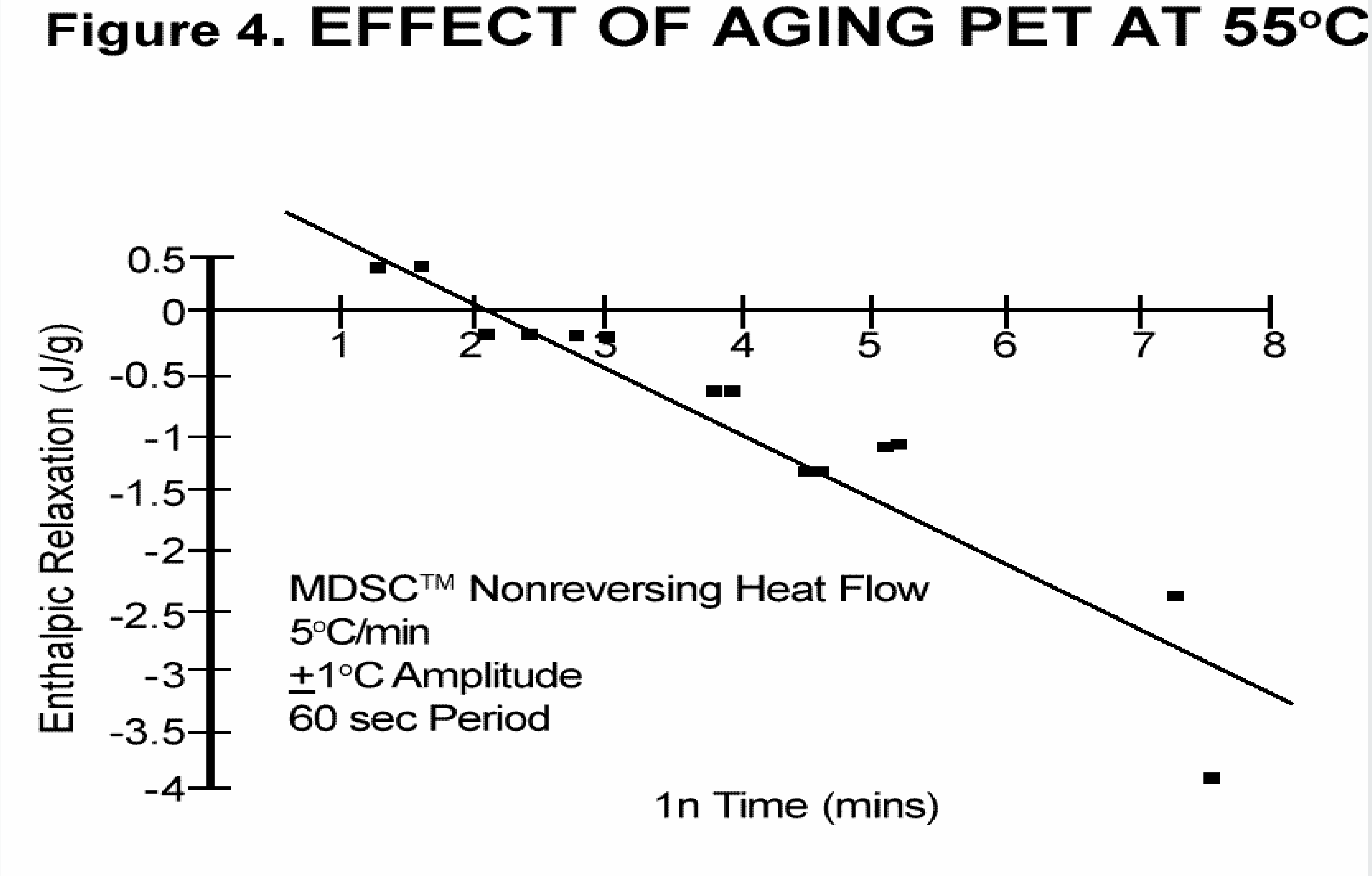
# PET焓鬆馳-TM-DSC 計算出絕對焓鬆馳值之圖譜



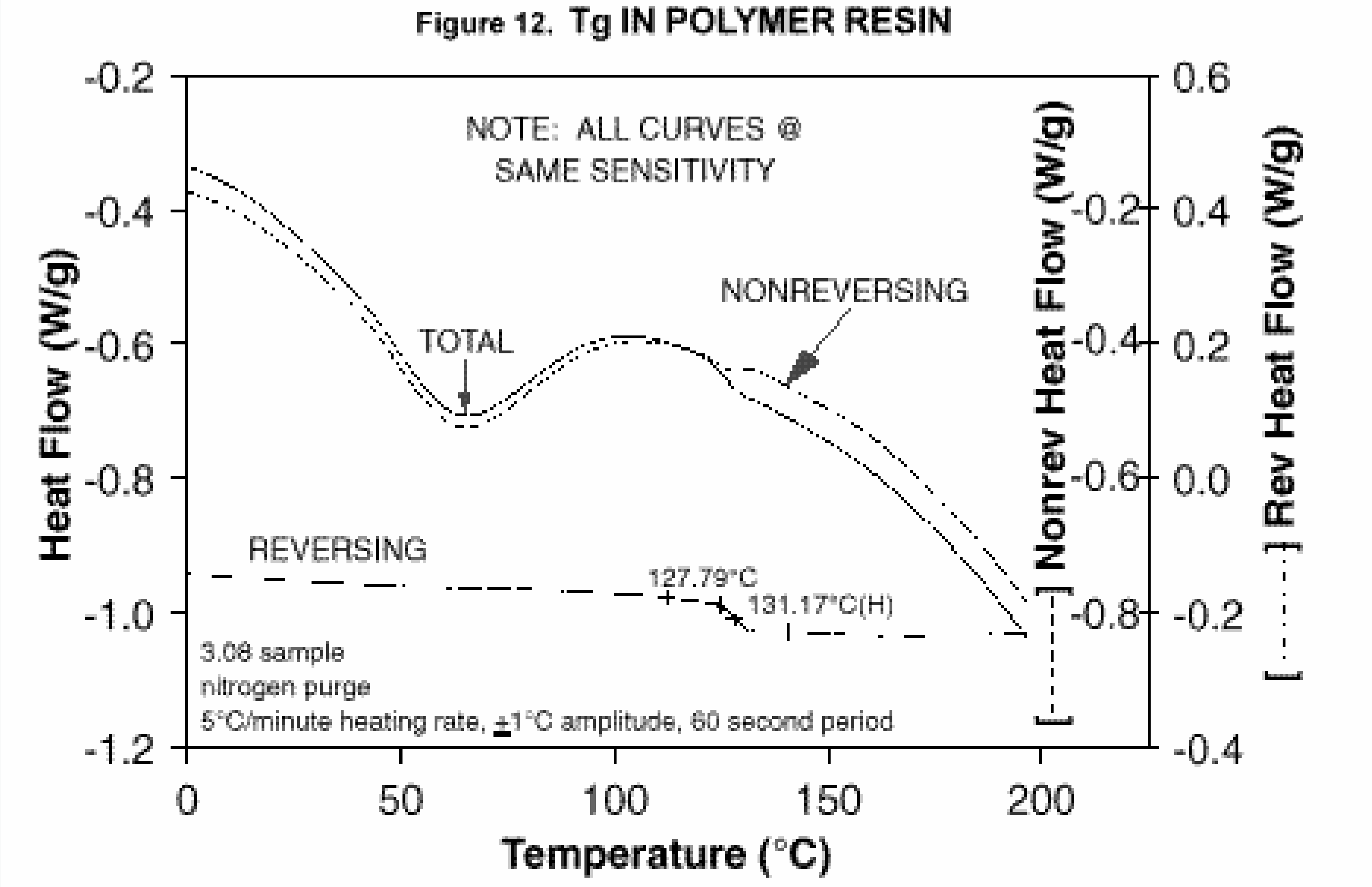
# PET焓鬆馳-不同老化時間使用TM-DSC 測得之圖譜



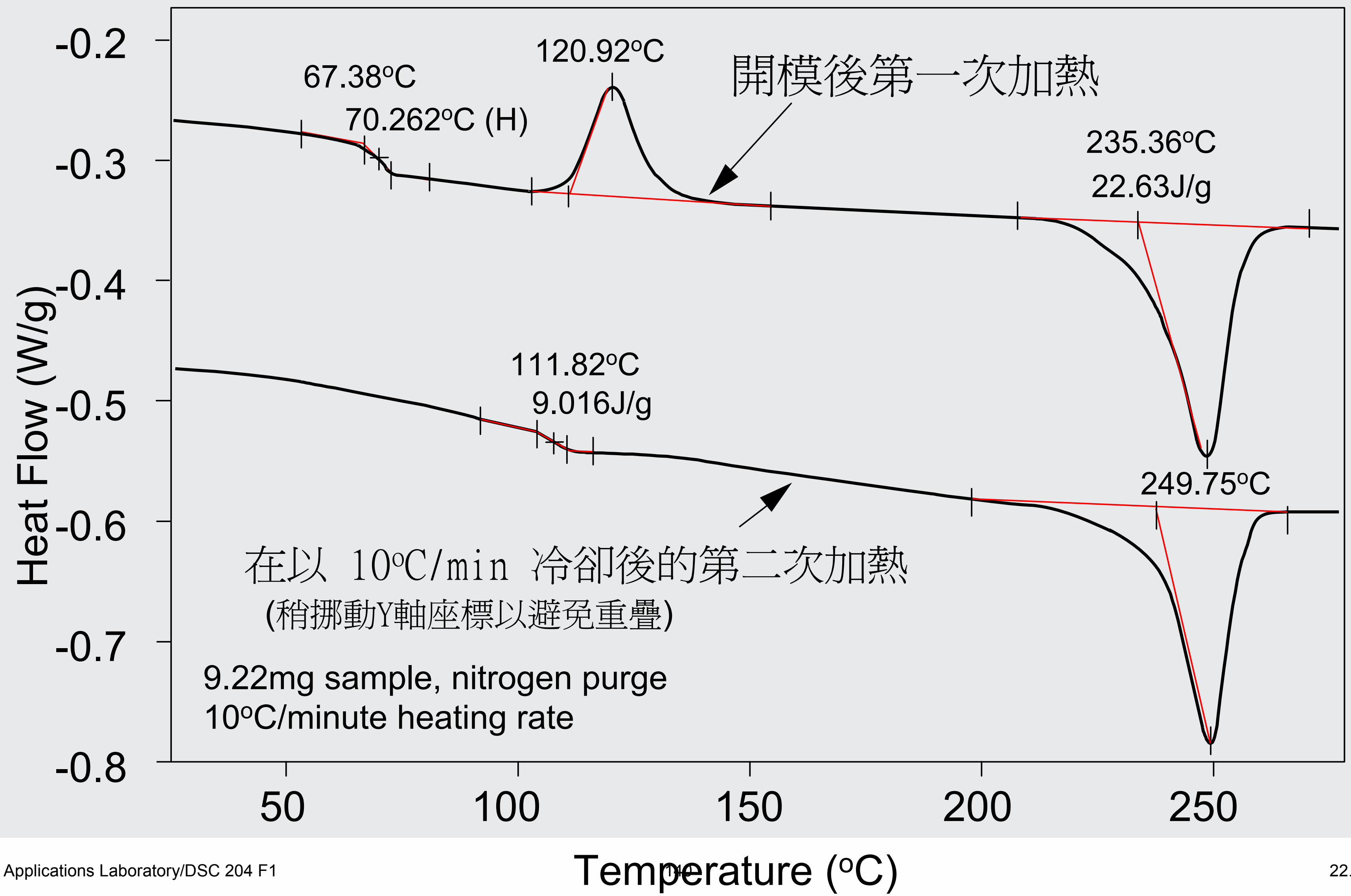
# PET焓鬆馳-不同老化時間所計算出檢量線圖譜



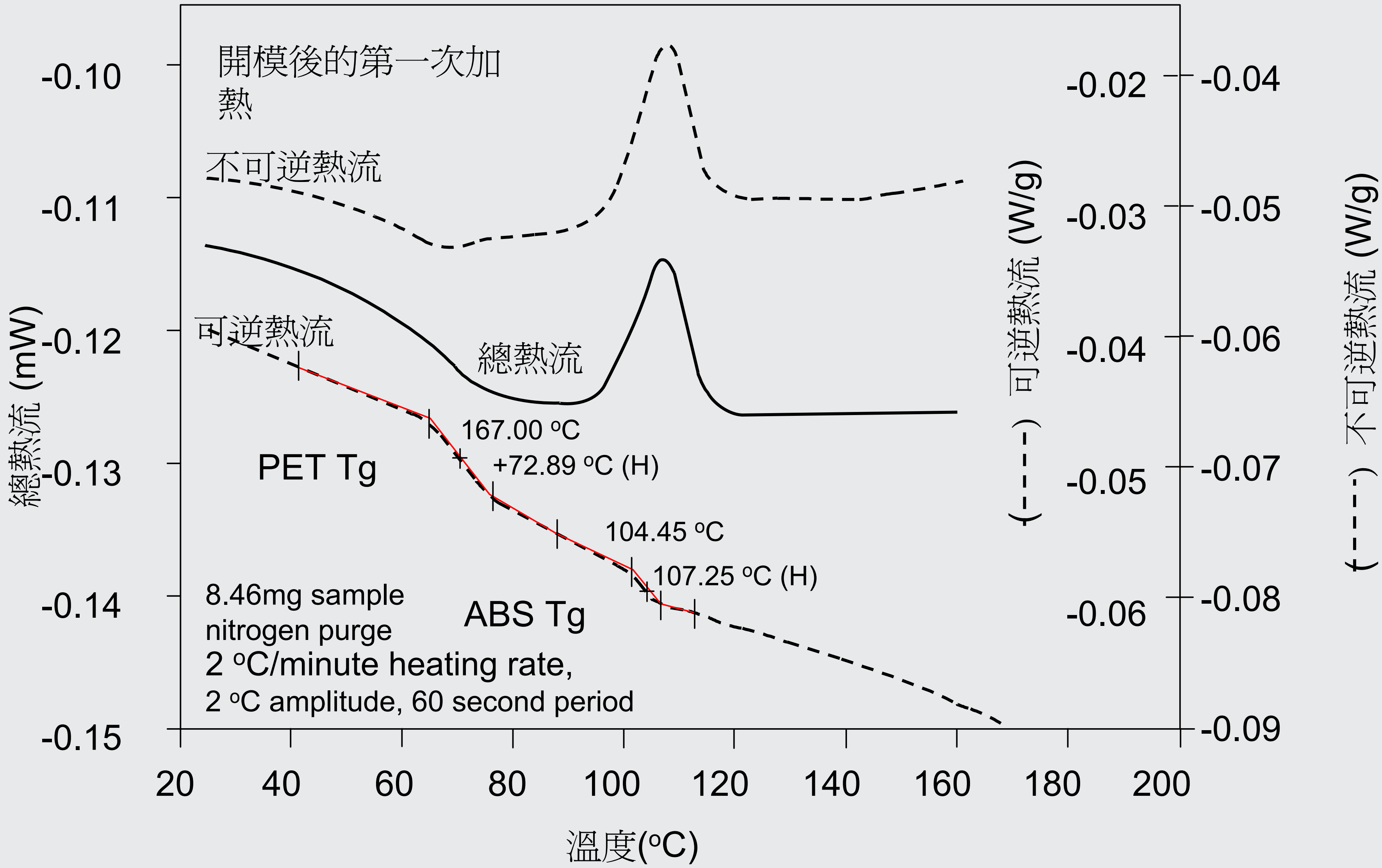
# TM-DSC 靈敏度增加-微弱相轉移測試



# PET/ABS聚攙物 - 傳統DSC的作法



# TM-DSC - PET/ABS聚攙物



# The DSC 204 *F1 Phoenix* System

**Flexible and robust**

**Optimized gas flow and cooling systems**

***Proteus*® Software with innovative features**

**Compliant with 21 CFR Part 11 (in 04/2006)**