

Audio test:



Termická analýza



01 Diferenciální Skenovací kalorimetrie (DSC)

Přednášející: Doc. Jiří Sopoušek

Obsah

- Heat flux DSC a Power compensation DSC
- Teorie
- Kalibrace
- Měření lat. Tepel a C_p
- Diskuse



ICTAC: DSC is the generic term for the following two measurement methods.

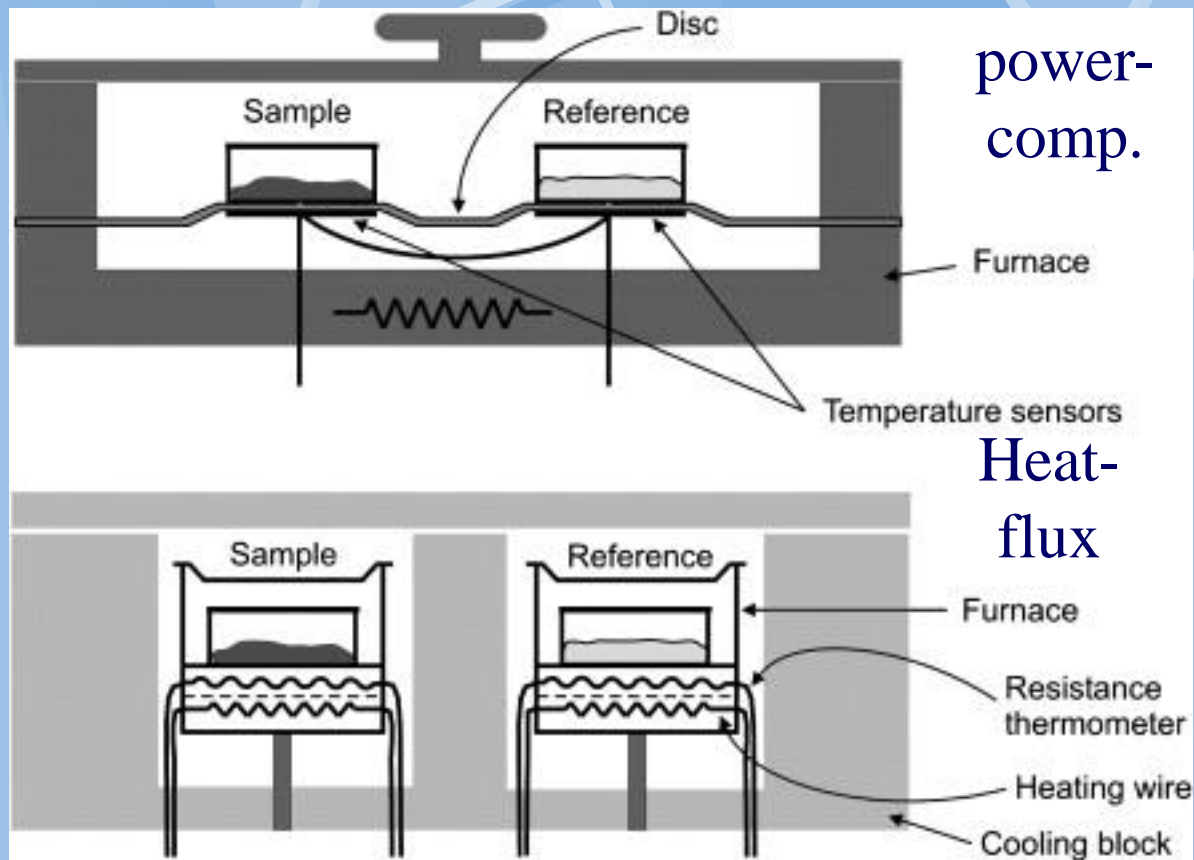
- Heat Flux DSCs

A technique in which the temperature of the sample unit, formed by a sample and reference material, is varied in a specified program, and the temperature difference between the sample and the reference material is measured as a function of temperature.

- Power Compensation DSC

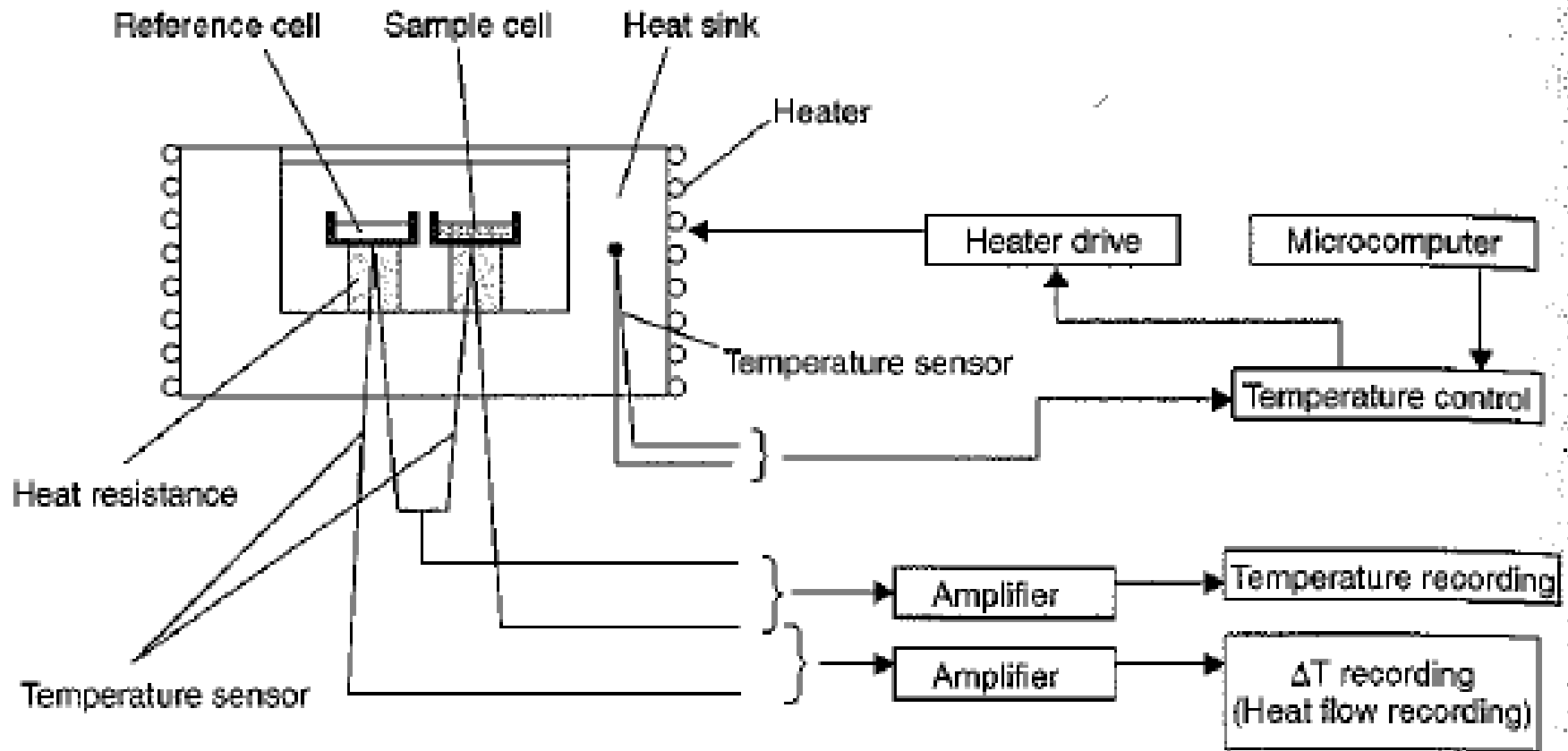
A technique in which difference of **thermal** energy that is applied to the sample and the reference material per unit of time is measured as a function of the temperature to equalize their temperature, while temperature of the sample unit, formed by the sample and reference material, is varied in a specified program.

Základní typy DSC: heat-flux a power compensation

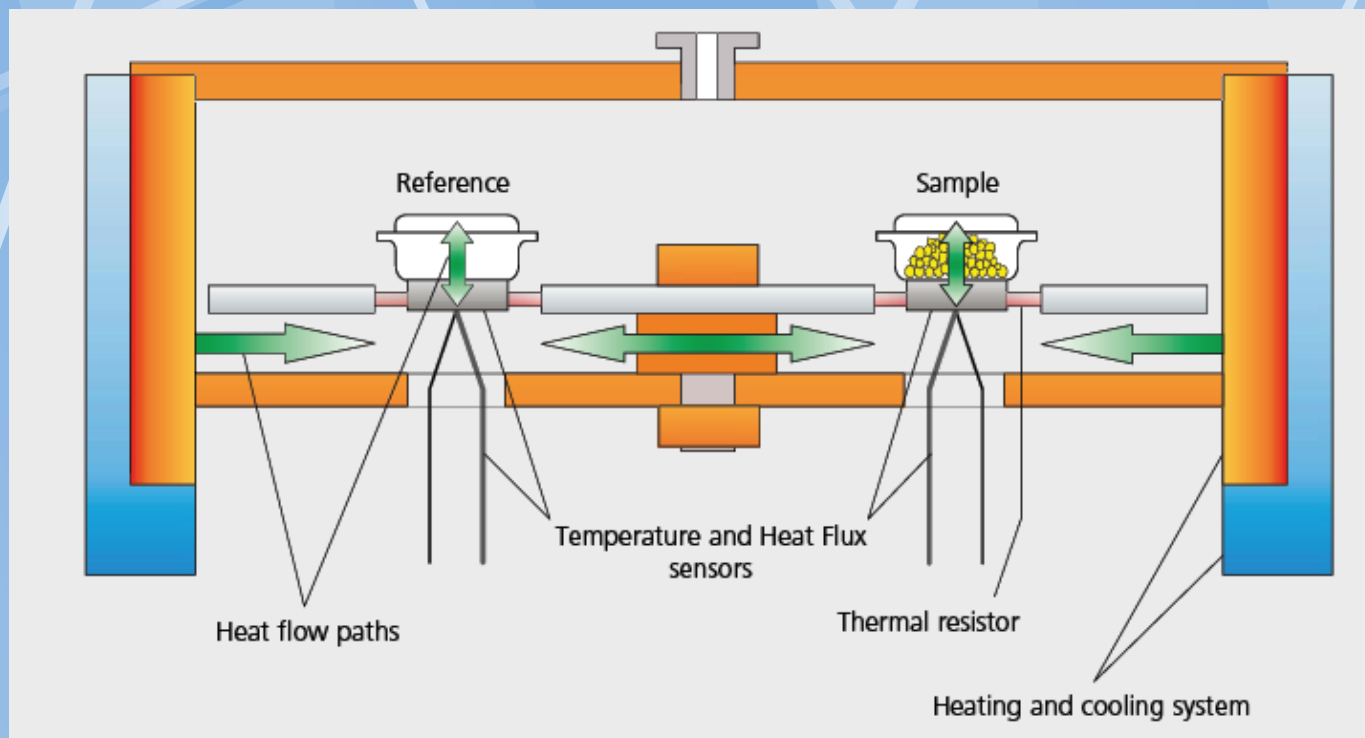


Rozdíly: cena, přesnost, periférie, výměna pecí,

hfDSC (heat-flux)

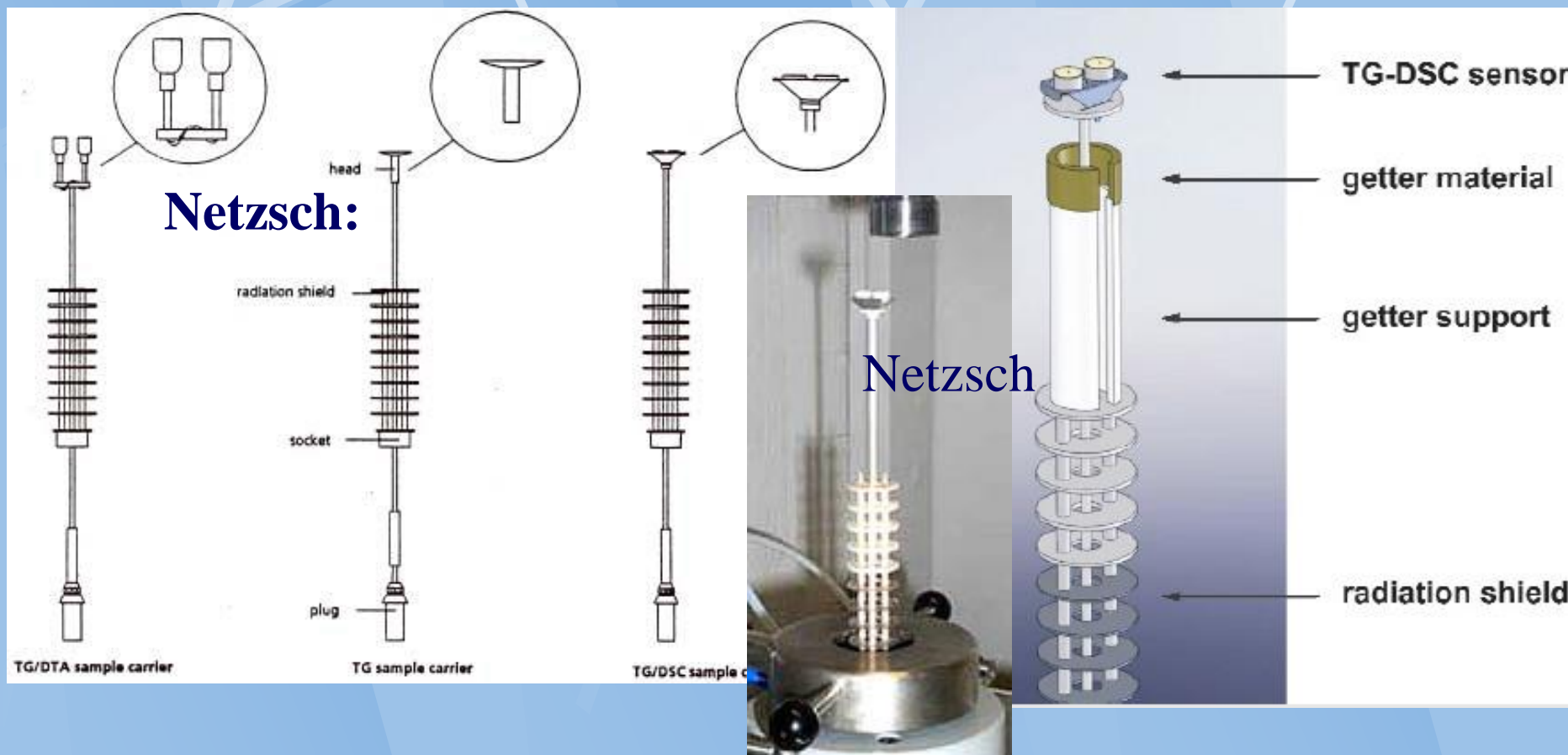


Držák hfDSC

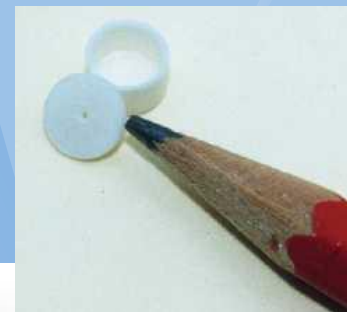


DSC Netzsch 204 Phoenix

Výměnné držáky hfDSC



Kelímky



Kelímky (ampule), lisovací kelímky Al.

Reakce s kelímkem, čištění kelímků,

**Na DSC signál má vliv: kontakt se dnem kelímku (1ks vzorku),
velikost vzorku, rychlost ohřevu, ..**

Kelímky

● Pan Type

- ↗ Aluminum
- ↗ Copper
- ↗ Gold
- ↗ Graphite
- ↗ Al Hermetic
- ↗ Al Alodined Hermetic
- ↗ Gold Hermetic
- ↗ High Volume (100μL)
- ↗ Al Solid Fat Index (SFI)
- ↗ Platinum

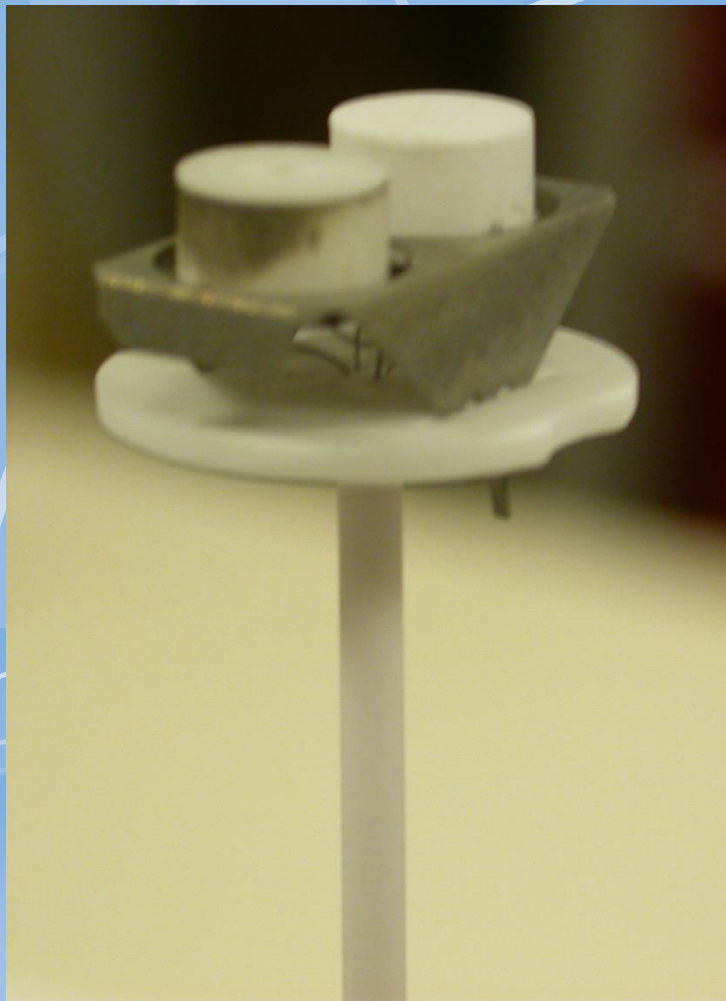
● Upper Temp Limit

- ↗ 600°C
- ↗ 725°C (in N₂)
- ↗ 725°C
- ↗ 725°C (in N₂)
- ↗ 600°C (3 atm.)
- ↗ 600°C (3 atm.)
- ↗ 725°C (6 atm.)
- ↗ 250°C (safety lid)
- ↗ 600°C (no cover)
- ↗ 725°C (no cover)

Použití kelímků

Materiál	Ano	Ne		Pozn.
Slinutý Al ₂ O ₃	Kovy	Oxidy		Levné, ne pro Ti
Pt	Anorganika, org. látky	Kovy		Drahé
Al	Organika	Kovy		Levné do 600stC
Cu				
Pf+Al ₂ O ₃	Kovy			Měření Cp
Fe				
Au	Biolog. materiál	Kovy		Spec. alikace, jinak nevhodné
grafit	Au,Ag			Pro nereaktivní kovy

Kelímky DSC – korundové kelímky



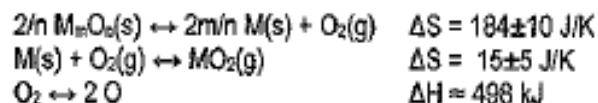
Víčka mj. chrání před
radiačními ztrátami
tepla!

Měřicí a referenční kelímek po měření vzorku AlNiZn (ANZ3)

{H ₂ O}	286
{H ₂ O ₂ }	94

$-\Delta H_f$ in kJ/mol for $\frac{1}{2}$ O₂

Li ₂ O	595	BeO	810
Li ₂ O ₂	318		



Na ₂ O	415	MgO	601
NaO ₂	261		
NaO ₂ *	130		
Na ₂ O ₂ *	256		

Stabilita oxidů

K ₂ O 361 KO ₂ 142 K ₂ O ₂ * 142	CaO 635	Sc ₂ O ₃ 623	Ti ₂ O ₅ 490 TiO ₂ 472 Ti ₂ O ₃ 507 TiO* 543	V ₂ O 456 VO 431 V ₂ O ₃ * 406 VO ₂ * 357 V ₂ O ₅ * 310	Cr ₂ O ₃ 376 CrO ₂ 293 CrO ₃ * 196	MnO 385 Mn ₃ O ₄ 346 Mn ₂ O ₃ * 319 MnO ₃ * 270	Fe _{0.95} O 264 Fe ₃ O ₄ 280 Fe ₂ O ₃ * 274	CoO 239 Co ₃ O ₄ 226
Rb ₂ O 330 Rb ₂ O ₃ 176	SrO 604 SrO ₂ 326	Y ₂ O ₃ 627	ZrO ₂ 547	NbO 410 NbO ₂ 399 Nb ₂ O ₅ * 380	MoO ₂ 294 MoO ₃ 248	TcO ₂ 216 TcO ₃ 180	RuO ₂ 150 RuO ₄ 46	RhO 92 Rh ₂ O ₃ * 119 RhO ₄ 96
Cs ₂ O 318 Cs ₂ O ₃ 188	BaO 581 BaO ₂ 318	La ₂ O ₃ 597	HfO ₂ 556	Ta ₂ O ₅ 408	WO ₂ 295 W ₃ O ₃ 284 WO ₃ * 281	ReO ₂ 213 ReO ₃ 205 Re ₂ O ₇ * 178	OsO ₂ * 147 OsO ₄ 97	IrO ₂ 111
Fr	Ra	Ac						

Ce ₂ O ₃ 599 CeO ₂ 594	Pr ₂ O ₃ 603 PrO ₂ 475	Nd ₂ O ₃ 602	Pm	Sm ₂ O ₃ 609
ThO ₂ 613	Pa	UO ₂ 541 U ₄ O ₉ 502 U ₂ O ₅ * 447 UO ₃ * 408	NpO ₂ 514	PuO ₂ 527

			B_2O_3 426	(CO_2) 197 (CO) 111	(N_2O_4) -10 (N_2O_5) -15	O ..	(OF_2) 18	Ne
			Al_2O_3 558	SiO_2 455	P_2O_3 273 P_2O_5 61 $P_2O_5^*$ 300	SO_2 151	(OCl_2) -81	Ar
NiO 240	Cu_2O 171 CuO 155	ZnO 348	Ga_2O 355 Ga_2O_3 363	GeO 212 GeO_2 363 GeO_2^* 290	As_2O_4 198 As_2O_5 185	SeO_2 112 Se_2O_5 82	Br	Kr
PdO 96 PdO^* 115	Ag_2O 30	CdO 258	In_2O_3 308	SnO 285 SnO_2 290	Sb_2O_3 240 SbO_2 290 SbO_2^* 227	TeO_2 161	I	Xe
PtO_2 -85	Au_2O_3 1	HgO 91	Ti_2O 166 Ti_2O_3 129	PbO 217 Pb_3O_4 179 PbO_2^* 137	Bi_2O_3 191	Po	At	Rn

EuO^* 590 Eu_2O_3 554	Gd_2O_3 607	Tb_2O_3 622 TbO_2 486	Dy_2O_3 621	Ho_2O_3 627	Er_2O_3 632	Tm_2O_3 629	Yb_2O_3 604	Lu_2O_3 625
Am_2O_3 585	Cm	Bk	Cf	Es	Fm	Md	No	Lw

Povlaky kelímků

Y₂O₃.....pro slitiny kovů , a Ti slitiny

BN....Al slitiny

Encapsulace

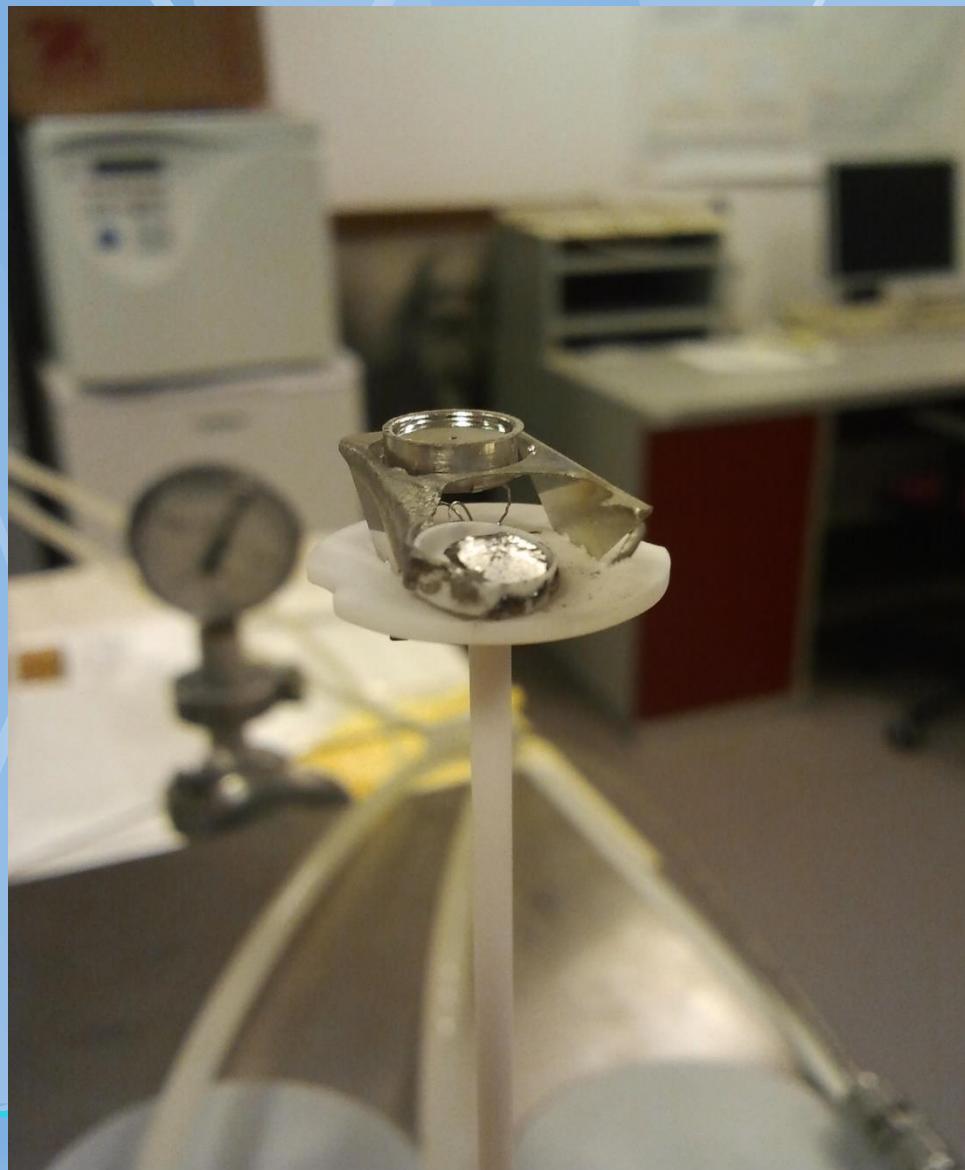


Organika, nano, ...

Špatná volba kelímků

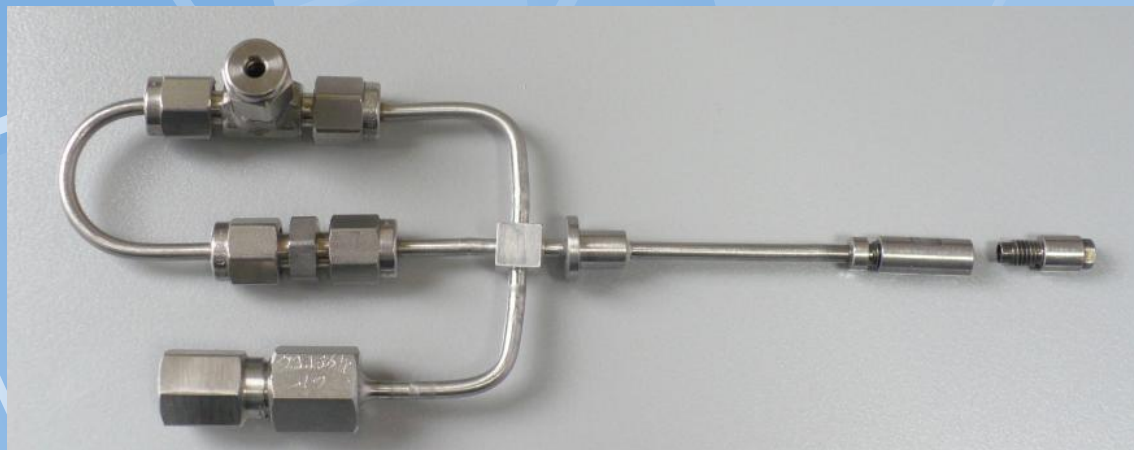
Včetně par

Pt nesnáší: Hg, Zn,
Mn,... (vznik
eutektik)



Plyny

Čištění plynů:
trapy, vymrazování
N₂, atd.



plyn	Použití	Pozn.
Synt. Vzduch	Oxidy, kinetika	Oxidace
Argon	Kovy slitiny	Inert
Dusík	Kovy, organika	Vznik nitridů
Helium	Speciální	Drahé
Redukční směsi N ₂ -H ₂	Kinetika	Pozor na termočlánky

	Thermal Conductivity at 300 K (W/mK)
Air	0.026
Ar	0.018
CO	0.025
CO ₂	0.017
H	0.182
He	0.151
N ₂	0.026
Ne	0.049
O ₂	0.027

pcDSC (kompensační DSC)

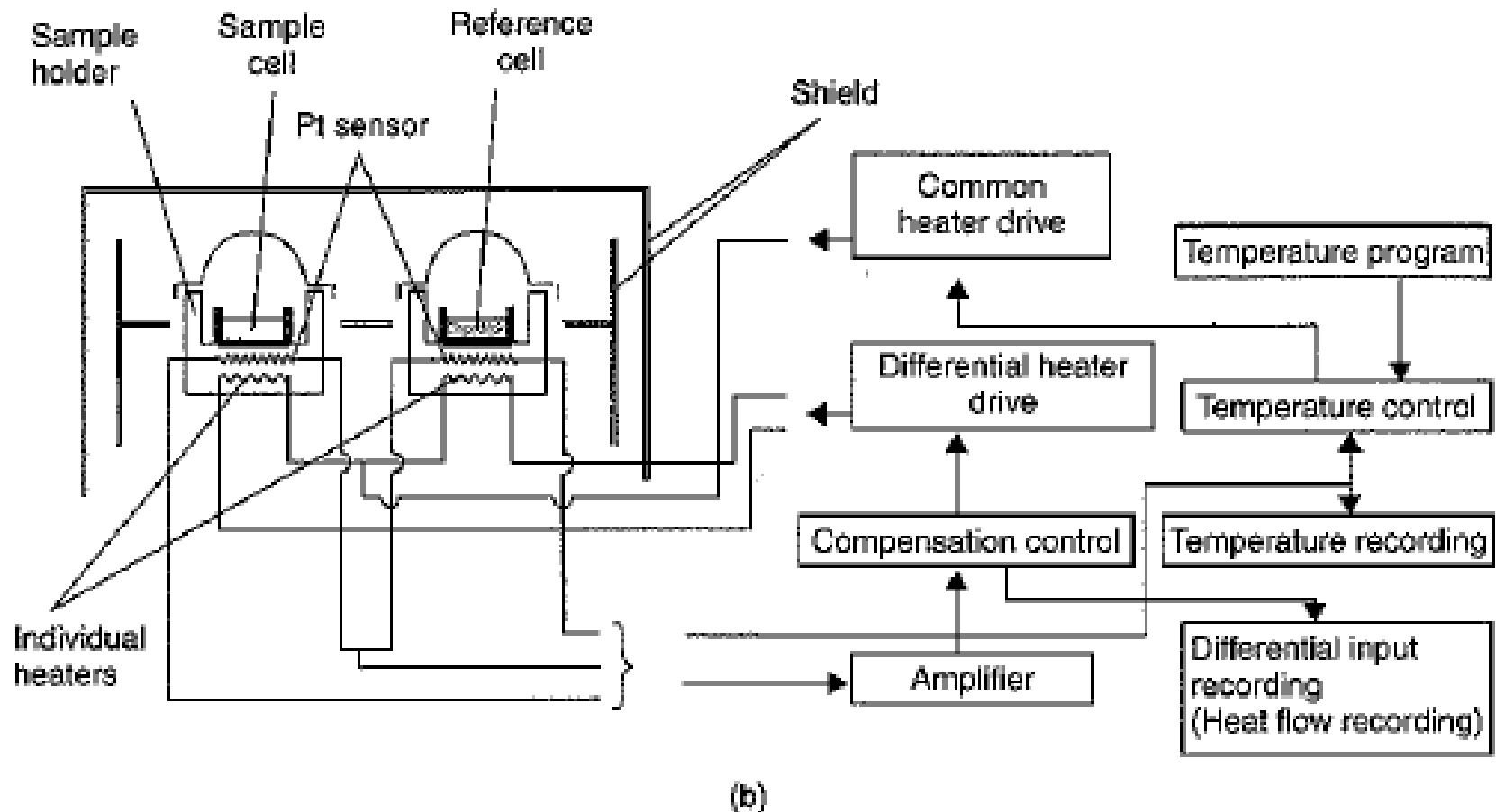


Figure 2 (a) Basic construction of heat-flux DSC (with copyright permission from Seiko Instruments Inc.). (b) Basic construction of power compensation DSC (with copyright permission from Seiko Instruments Inc.)

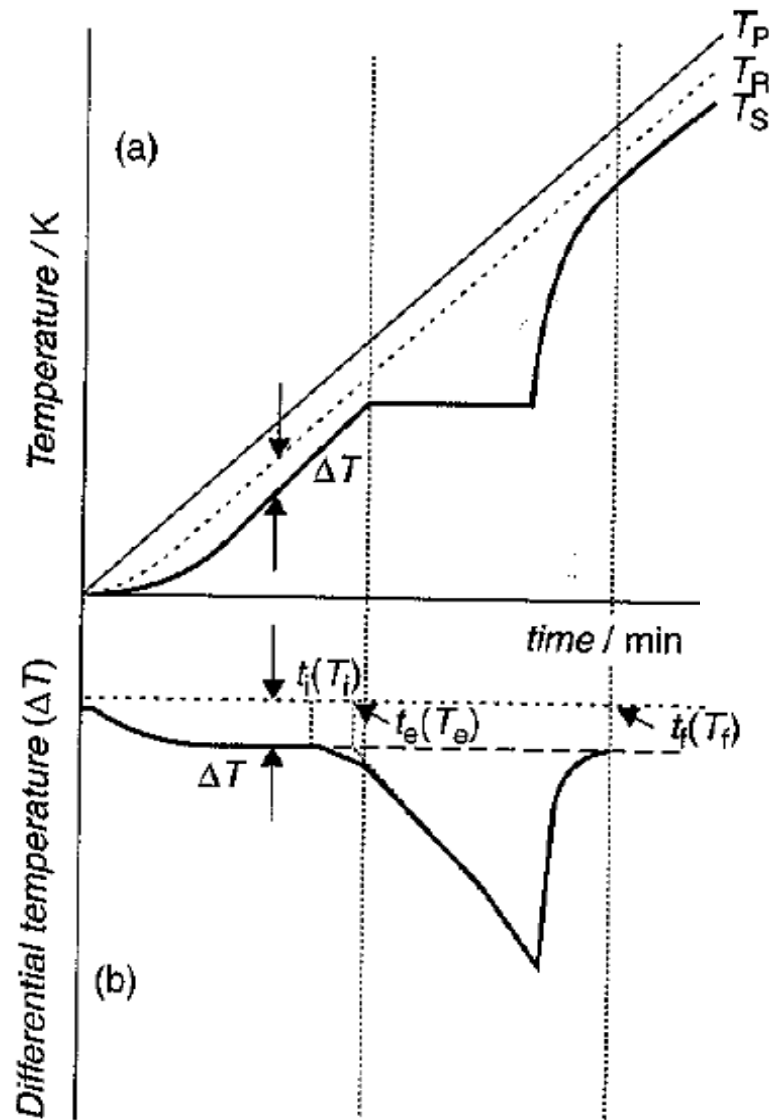
Praktická realizace pcDSC

Velmi podobné hfDSC

Záznam DSC

$$dT_p/dt = \text{konst.}$$

Stacionární
stav



Differential Scanning Calorimetry (DSC)

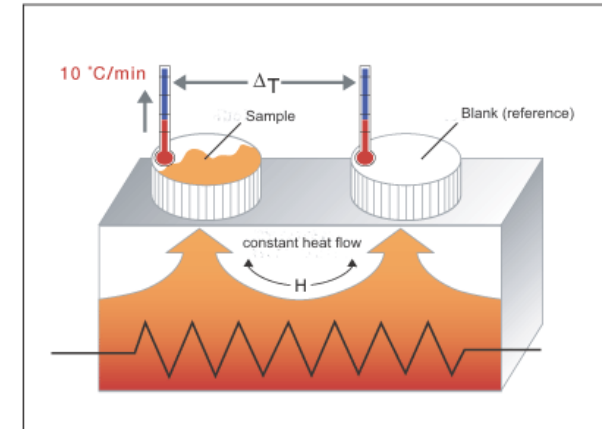
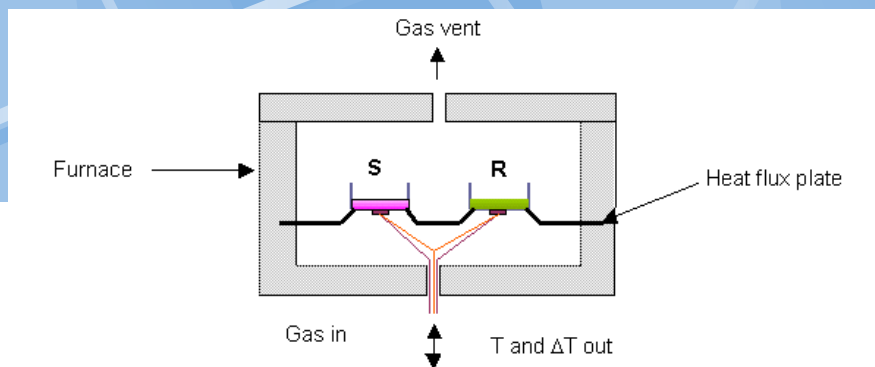


Figure 1 Basic principle of DTA and DSC: (a) change in temperature of reference T_R and sample T_S with increasing furnace temperature T_P ; (b) typical signal output converted to differential temperature ΔT with passage of time

Teorie DSC

$$dT_p/dt = \text{např. } 0\text{K/min}$$



Sample S

Reference R

Kelímek
sample CS

Kelímek ref.
CR

Tepelný odpor
mezi CS a P

Tepelný odpor
mezi CR a P

Tepelný odpor
mezi CS a S

Tepelný odpor
mezi CR a R

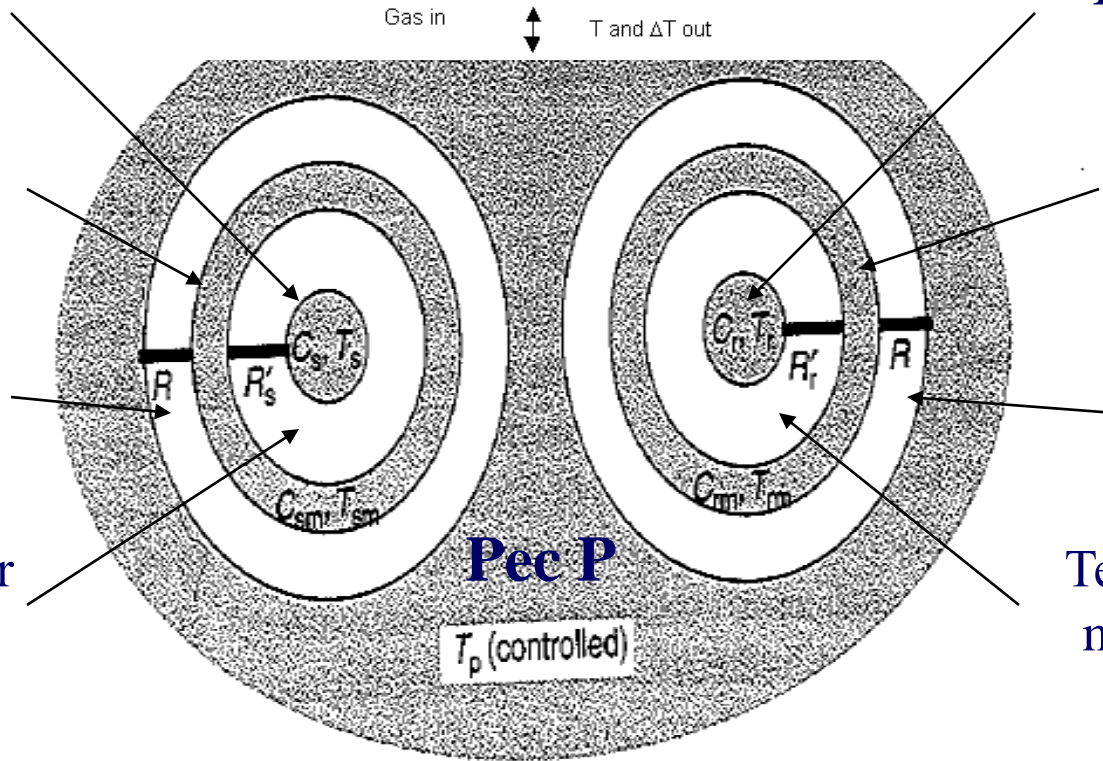


Figure 1 Mraw's model for classical DTA, power-compensated DSC and heat-flux DSC

Teplo z pece do vzorku:

$$dq_S/dt = \frac{1}{R}(T_P - T_S)$$

Současně ve stc stavu $dT=dT_P$:

$$dq_S/dt = [C_{CS}(T) + C_S(T)] dT_P/dt$$

$G_T=1/R$...tepelná vodivost (C,C,R)

R... tepelný odpor mezi pecí a vzorkem ($R+R_S$)

C_{CS}, C_S ...tepelná kapacita cely se vzorkem a vzorku

Teplo z pece do reference

$$\begin{aligned} dq_R/dt &= \frac{1}{R}(T_P - T_R) \\ &= [C_{CR}(T) + C_R(T)] dT_P/dt \end{aligned}$$

Rozdíl tepelných toků:

$$\begin{aligned} dq_S/dt - dq_R/dt &= -\frac{1}{R}(T_S - T_R) \\ &= \{[C_{CS}(T) - C_{CR}(T)] + [C_S(T) - C_R(T)]\} dT_P/dt \quad (4) \end{aligned}$$

Je-li hmotnost a kapacita cely vzorku a cely pro referenci stejná. Tj
($C_{CS}(T) = C_{CR}(T)$),

Pak:

$$\frac{1}{R}(\Delta T) = [C_S(T) - C_R(T)] dT_P/dt$$

kde

$$\Delta T \equiv T_R - T_S$$

$$\frac{1}{R}(\Delta T) = [C_S(T) - C_R(T)]dT_P/dt$$

hfDSC

Současně platí pro rozdíl tepla přijímaného vzorkem a referencí:

$$d\Delta q/dt \equiv dq_S/dt - dq_R/dt = [C_S(T) - C_R(T)] dT_P/dt$$

R (tep. Odpor mezi pecí a vzorkem, resp. referencí) je pro DTA obtížně definovatelné, pro hfDSC lépe (platinová ploška tvoří receptor tepla z pece) proto je hfDSC vhodná pro kvantitativní měření.

$d\Delta q_m/dt$ je měřený signál hfDSC

Power comp. DSC:

Rozdíl v teple potřebný pro $T_R = T_S$ poskytuje dodatečný ohřev ($d\Delta q_m/dt = dUI$). pcDSC má proto předpoklady být ještě přesnější.

Ale protože i u pcDSC za konst. ohřevu jsme v dynamické a nikoliv v termodynamické rovnováze je nutno zavést jistou přístrojovou korekci $K(T)$:

$$d\Delta q/dt = K(T)d\Delta q_m/dt = [C_S(T) - C_R(T)] dT_P/dt$$

kde $d\Delta q_m/dt$ = „dodatečný výkon“ je měřený
signál pcDSC

Experiment a měření

Tepelná kapacita:

$C_R=0$...lze použít prázdný kelímek

$$d\Delta q/dt = K(T)d\Delta q_m/dt = [C_S(T) - C_R(T)] dT_P/dt$$

dopočteme $C_S = m_s c_s^g = n_s c_s^n$. Zjistíme c_s^n resp. c_s^g
(není ale přesné).

Nutno vylepšit měřením standardu: korund.

Měření C_p pomocí hf I pc DSC

- 1. Cyklus: prázdné kelímky CR a CS (tzv. baseline)
- 2. Cyklus: prázdný kelímek CR a CS s korundem Al_2O_3 .
- 3. Cyklus: prázdný kelímek CR a CS se vzorkem.

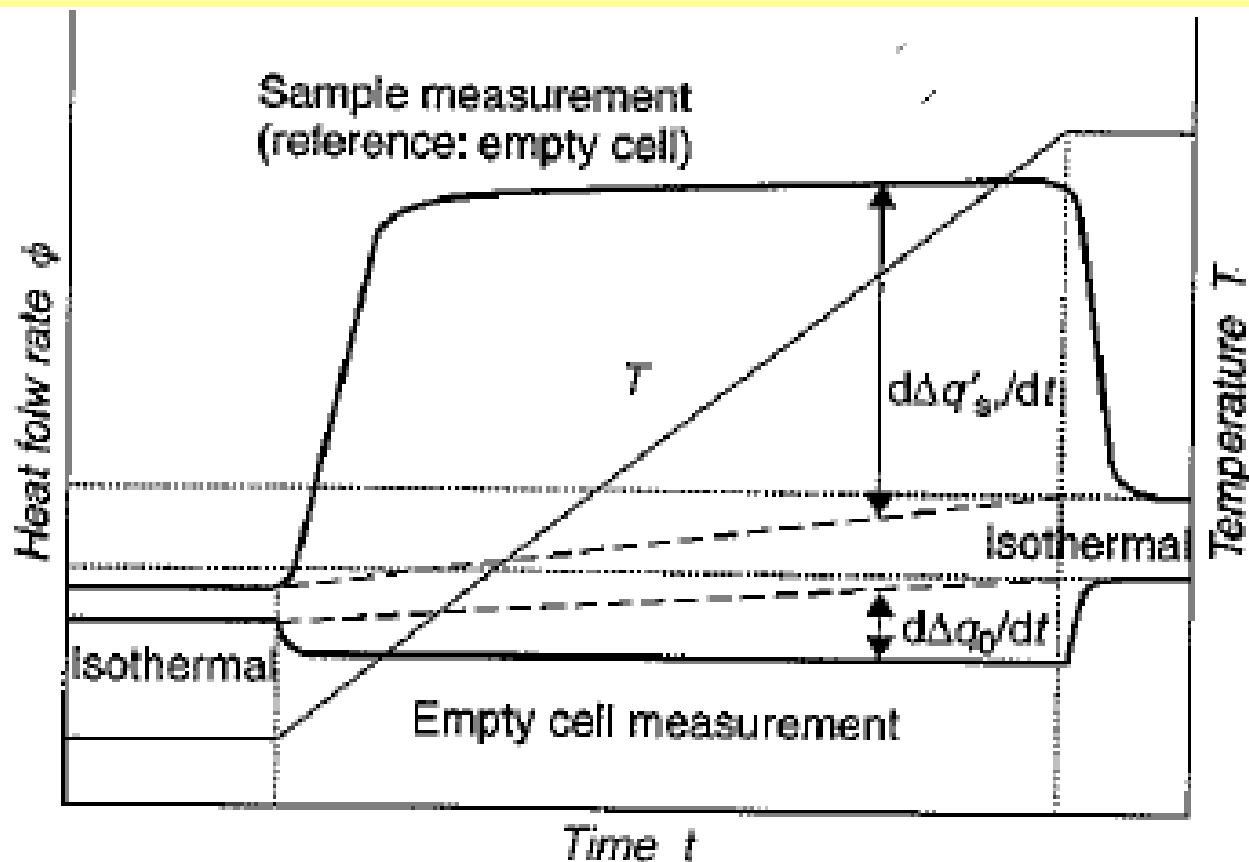


Figure 3 Schematic diagram of DSC output for heat capacity measurement

Cp alfa korundu

Table 1 Heat capacity of α -alumina (in $\text{J K}^{-1} \text{g}^{-1}$)

	$70 < T/\text{K} < 300$	$290 < T/\text{K} < 2250$
a_0	3.63245×10^{-2}	-5.81126×10^{-1}
a_1	-1.11472×10^{-3}	8.25981×10^{-3}
a_2	-5.38683×10^{-6}	-1.76767×10^{-5}
a_3	5.96137×10^{-7}	2.17663×10^{-8}
a_4	-4.92923×10^{-9}	-1.60541×10^{-11}
a_5	1.83001×10^{-11}	7.01732×10^{-15}
a_6	-3.36754×10^{-14}	-1.67621×10^{-18}
a_7	2.50251×10^{-17}	1.68486×10^{-22}

$$C_{\text{st}} = \sum_{n=0}^7 a_n T^n$$

Výhodnocení C_p :

Prázdné kelímky (baseline):

$$[C_{CS}(T) - C_{CR}(T)] dT_P/dt = K_\phi(T) d\Delta q_0/dt \quad (a)$$

Prázdný CR a CS se vzorkem:

$$\{C_S(T) + [C_{CS}(T) - C_{CR}(T)]\} dT_P/dt = K_\phi(T) d\Delta q_S/dt \quad (b)$$

Odečet (b)-(a):

$$C_S(T) dT_P/dt = K_\phi(T) d\Delta q'_S/dt \quad (c) \quad \text{where } d\Delta q'_S/dt \equiv d\Delta q_S/dt - d\Delta q_0/dt.$$

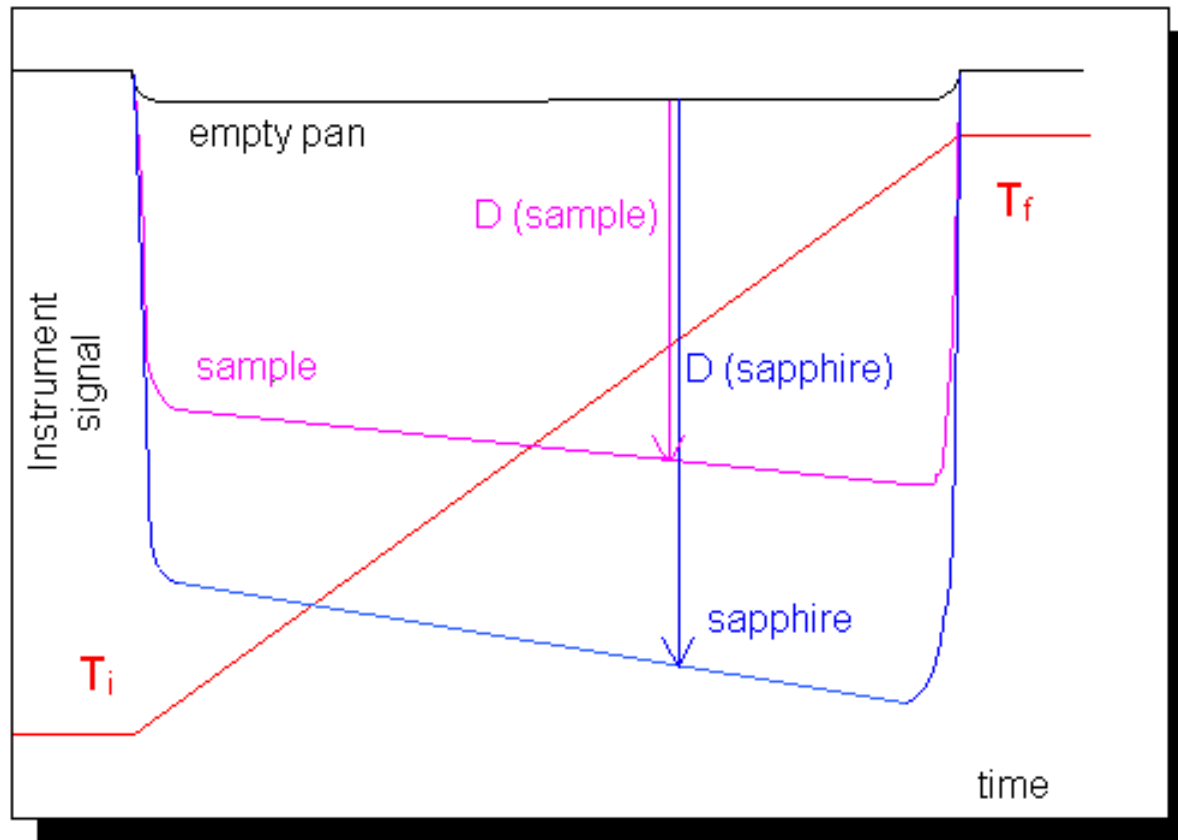
Podobně pro prázdný CR a CS se standardem:

$$C_{RM}(T) dT_P/dt = K_\phi(T) d\Delta q'_{RM}/dt \quad (d) \quad d\Delta q'_{RM}/dt \equiv d\Delta q_{RM}/dt - d\Delta q_0/dt.$$

Podělením (c) / (d) se zbavíme $K_\phi(T)$:

$$C_S(T) = [d\Delta q'_S/d\Delta q'_{RM}] \cdot C_{RM}(T)$$

hf DSC Cp měření



$$\frac{D(\text{sample})}{D(\text{sapphire})} = \frac{C_p(\text{sample}) \times \text{mass of sample}}{C_p(\text{sapphire}) \times \text{mass of sapphire}}$$

Cp pro PET

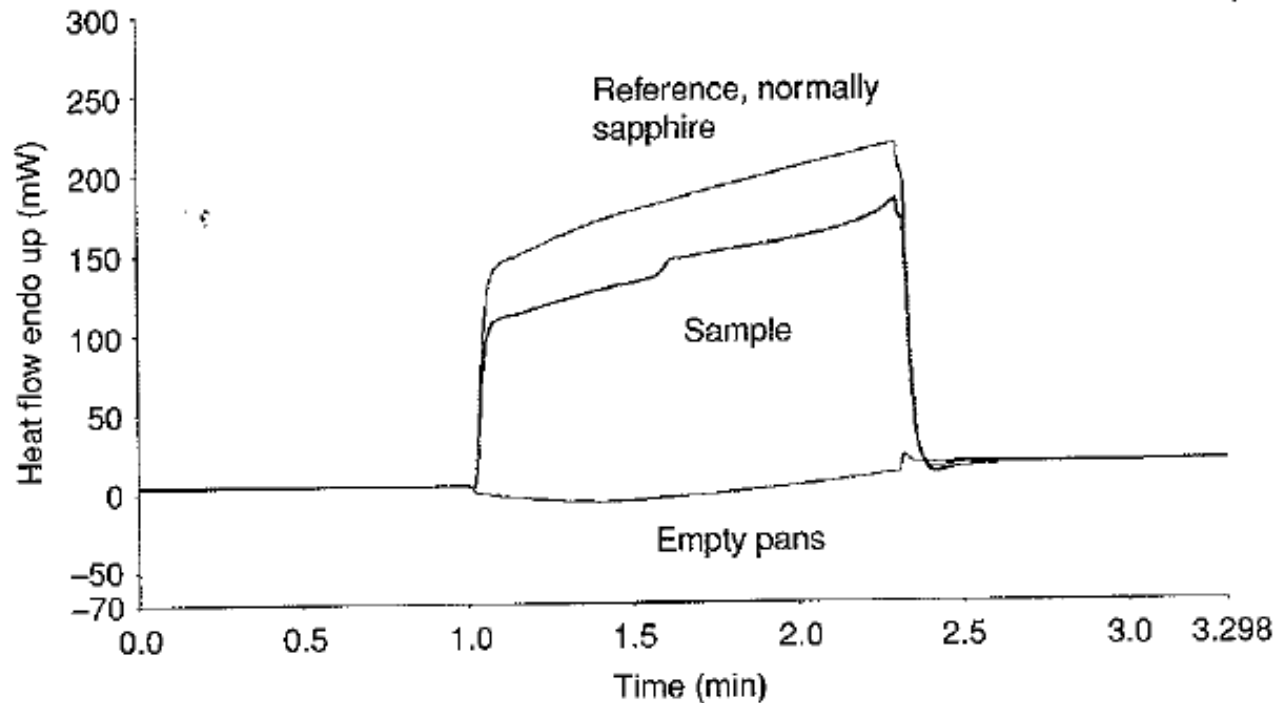


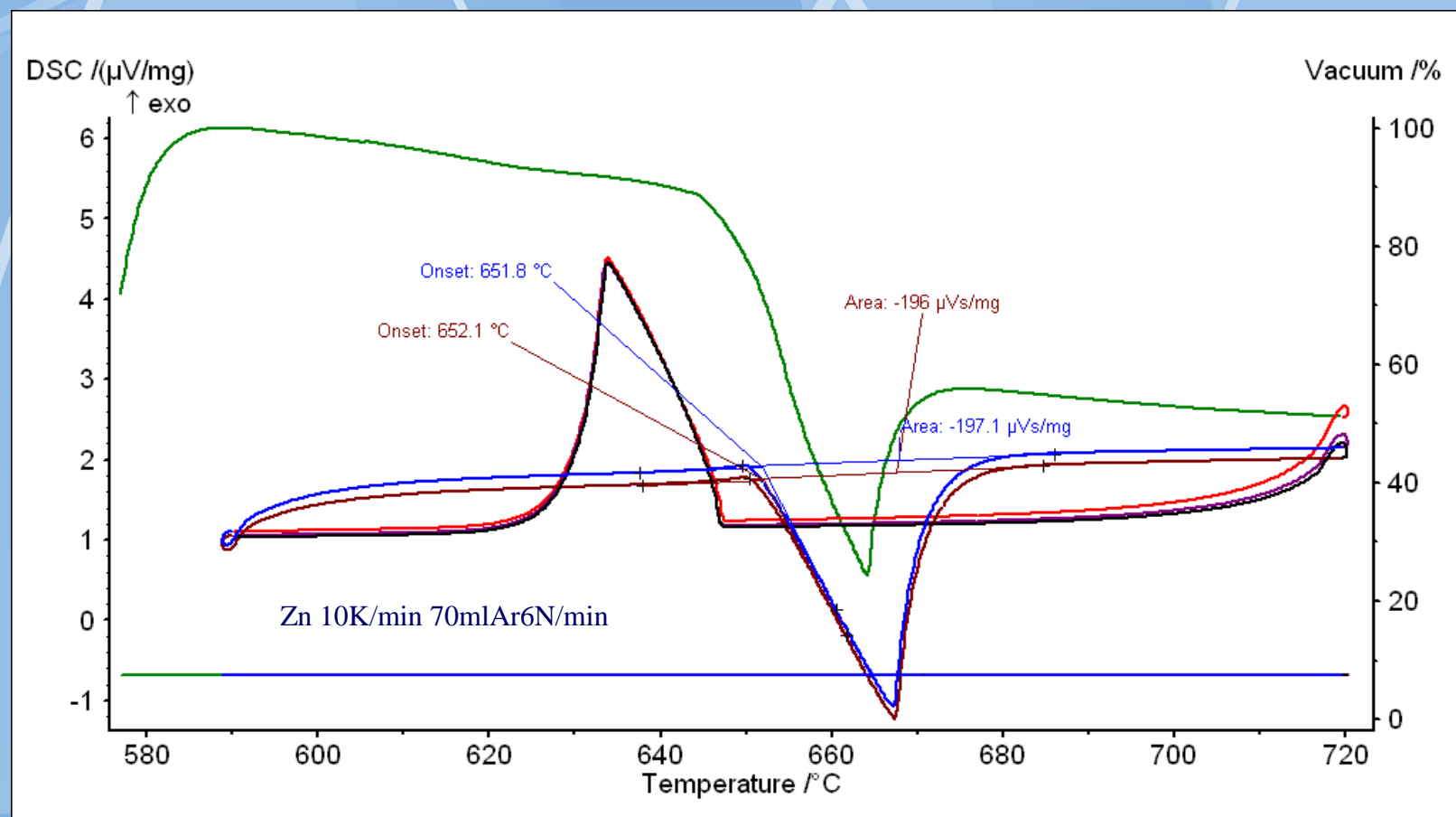
Figure 1.1 Heat capacity of PET obtained using fast scanning techniques showing the three traces required for subtraction. The height of the sample compared to the empty pan is divided by the scan rate and the mass of sample to obtain a value for C_p . This is referenced against a known standard such as sapphire for accuracy. If small heating steps of, for example, 1°C are used the area under the curve can be used to calculate C_p . This calculation is employed as an option in stepwise heating methods.

Měření latentních tepel FT

Fázová transformace

$$\Delta_{\text{trs}} H = K_{\phi}(T)A$$

A...plocha peaku



Exo – Endo (změna entalpie ΔH)

Process	Exotherm	Endotherm
Solid-solid transition	*	*
Crystallisation	*	
Melting		*
Vaporisation		*
Sublimation		*
Adsorption	*	
Desorption		*
Desolvation (drying)		*
Decomposition	*	*
Solid-solid reaction	*	*
Solid-liquid reaction	*	*
Solid-gas reaction	*	*
Curing	*	
Polymerisation	*	
Catalytic reactions	*	

Exo -
Endo
↑↓

Kalibrace

Pro konstantní rychlost ohřevu, plyn, držák,
termočlánek, kelímky, víčka,atd.

- Teplota (známe body F transformací, $T_{tr}=T_m+\Delta T(T)$
čisté kovy)
- Entalpie (plocha peaku, čisté kovy) $K_\phi(T)$
- Rychlost tepelného toku (korundový
standard)

Kalibrace na teplotu

Table 1.1 Commonly used standards and reference materials

Standard	Melting point (°C)	Heat of fusion (J/g)
Indium	156.6	28.42
Tin	231.9	
Lead	327.5	
Zinc	419.5	
K ₂ SO ₄	585.0	
K ₂ Cr ₂ O ₇	670.5	
Substance	Transition	Transition temperature (°C)
Cyclopentane	Crystal	−151.16
Cyclopentane	Crystal	−135.06
Cyclohexane	Crystal	−87.06
Cyclohexane	Melt	6.54
<i>n</i> -Heptane	Melt	−90.56
<i>n</i> -Octane	Melt	−56.76
<i>n</i> -Decane	Melt	−29.66
<i>n</i> -Dodecane	Melt	−9.65
<i>n</i> -Octadecane	Melt	28.24

Kovy

Anorg. Soli

Organika

Magn. Látky.

FT (eutektika,
peritektika)

Čisté kovy

Material	Tm (°C)	Hf (J/g)
Mercury	-38.8344	11.469
Gallium	29.7646	79.88
Indium	156.5985	28.62
Tin	231.298	7.170
Bismuth	271.40	53.83
Lead	327.462	23.00
Zinc	419.527	108.6
Aluminium	660.323	398.1

+ Ag,
Au,
Ni

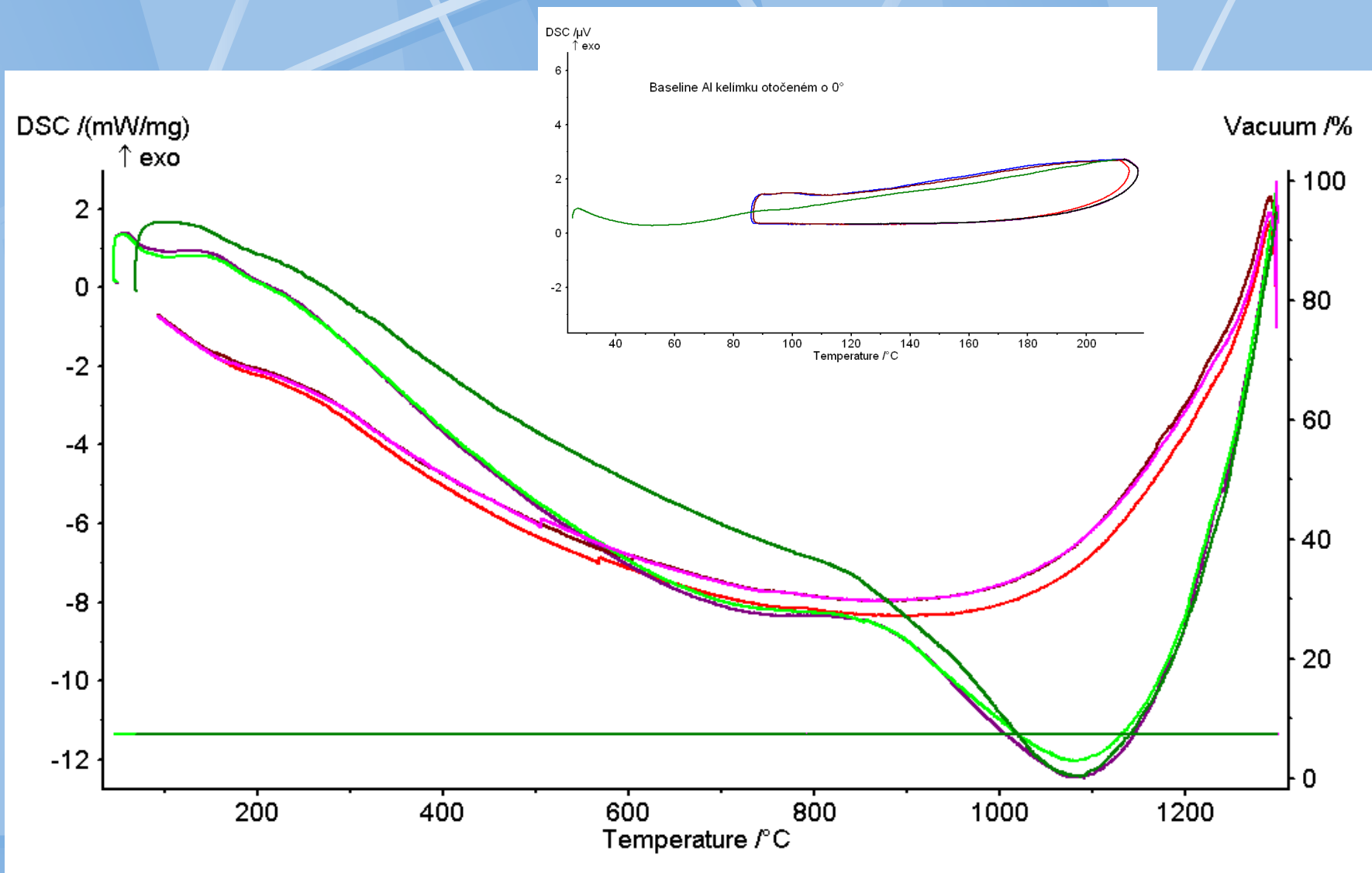
Další čisté kovy a eutektika

Table 2 -Fixed Point Temperatures for Calibration [69Bar, 90Pre, 96Bed]

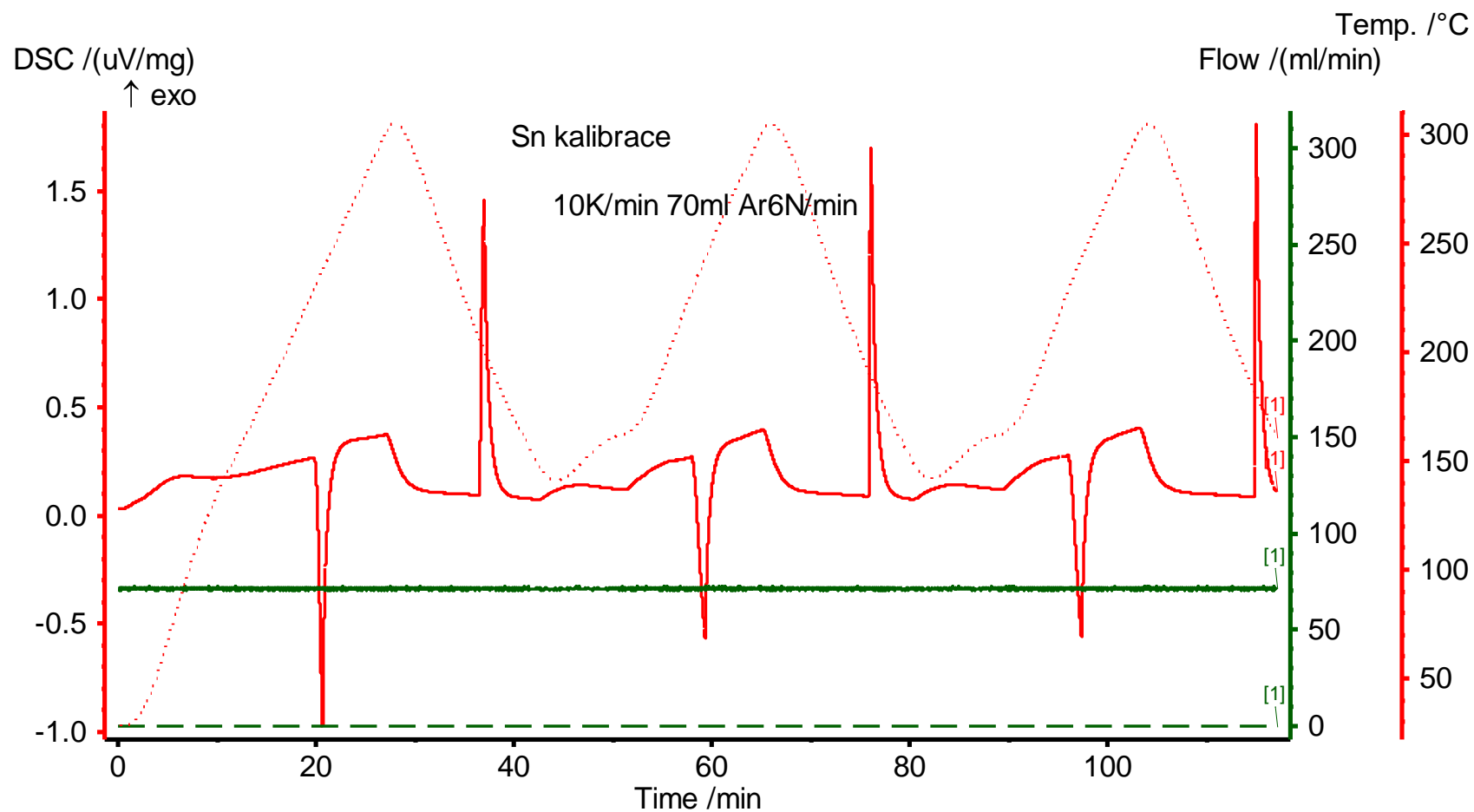
Substance	T/°C	Ref.
In	156.5985	90Pre
Sn	231.928	90Pre
Bi	271.402	96Bed
Cd	321.069	96Bed
Pb	327.462	96Bed
Zn	419.527	90Pre
Sb	630.628	96Bed
Al	660.323	90Pre
Ag - 28.1 % Cu*	779.63	96Bed
Ag	961.78	90Pre
Au	1064.18	90Pre
Cu	1084.62	90Pre
Ni	1455	69Bar
Co	1494	69Bar
Pd	1554.8	96Bed

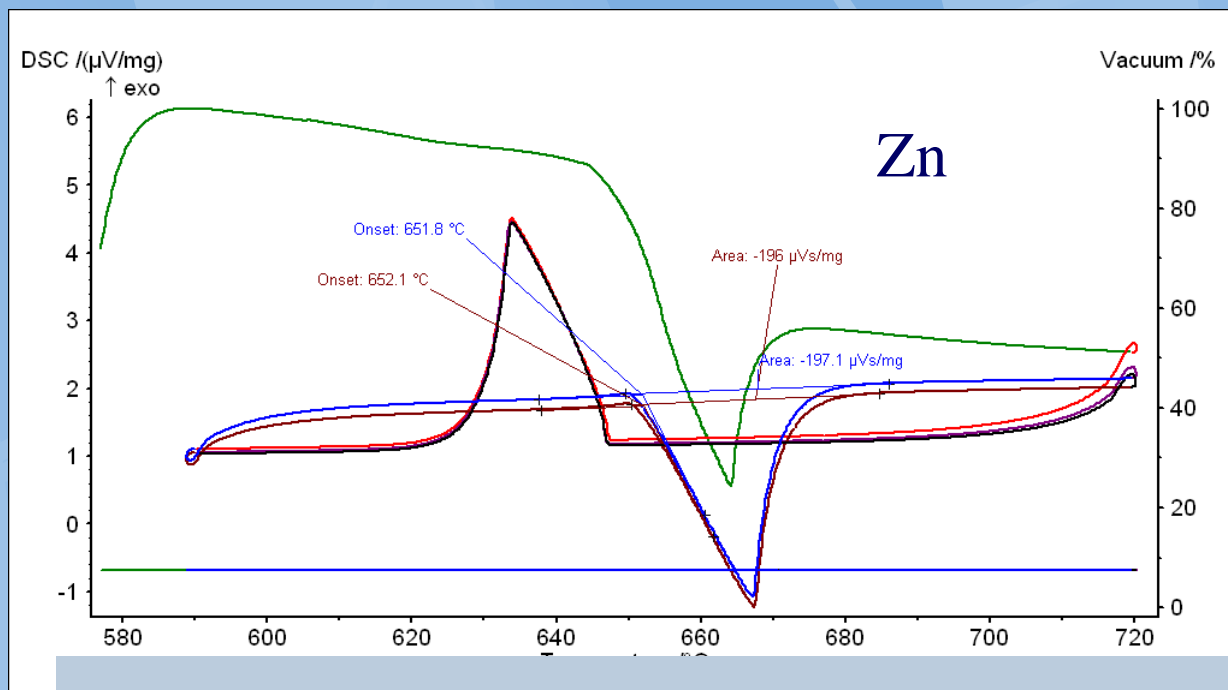
*Eutetic composition. In this document, the % symbol will refer to percentage by mass.

Baseline s prázdnými kelímký



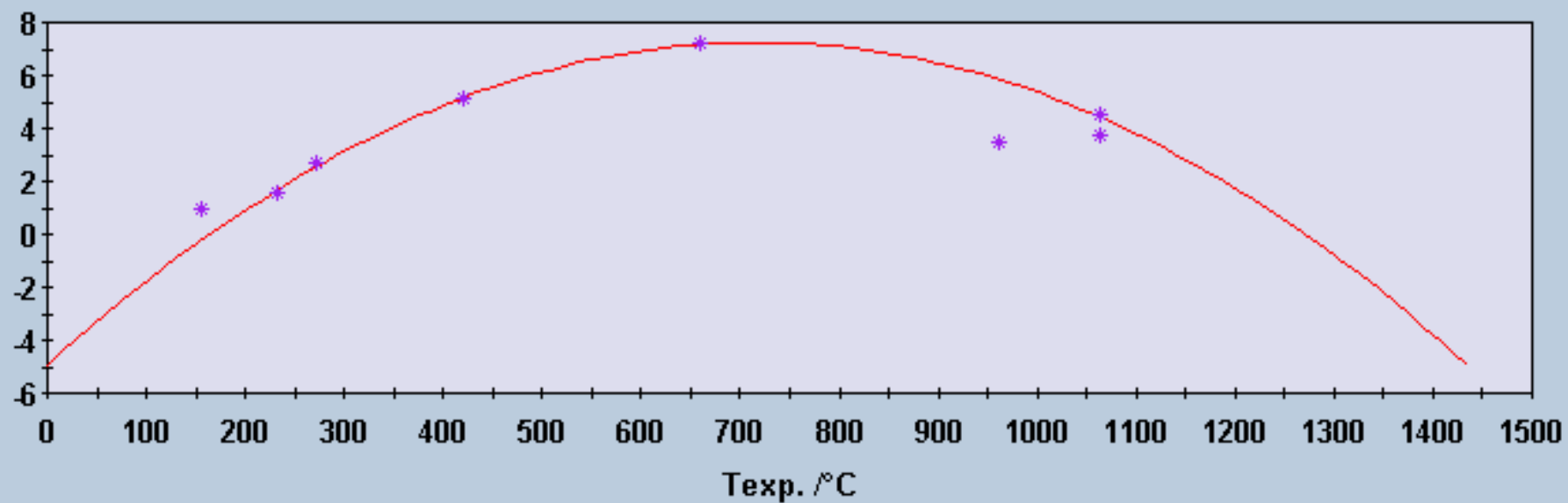
hfDSC kalibrace





Kalibrace na
teplotu

T_{nom.} - T_{exp.} °C



Kalibrace hfDSC na citlivost

Známy standard(In):

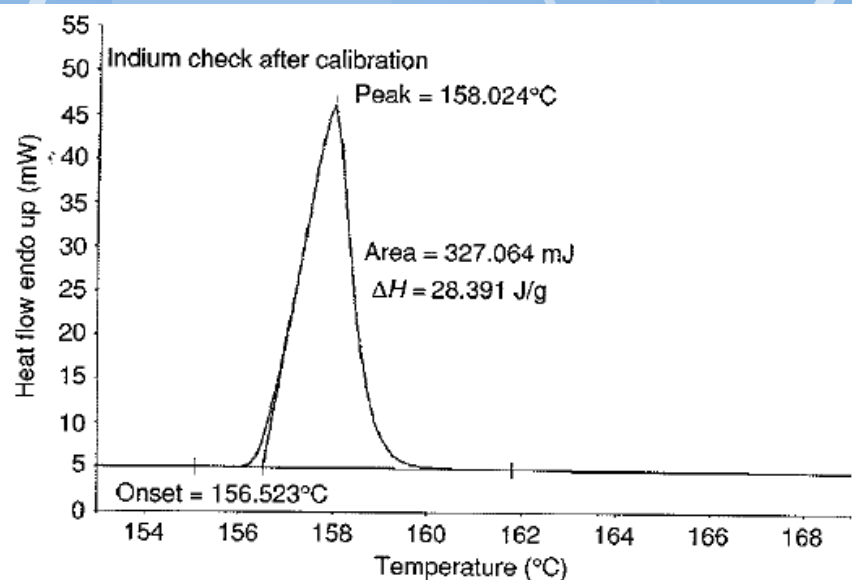


Figure 1.4 Indium run as a check after a calibration procedure has been completed showing the onset calculation for melting point, and area calculation for heat of fusion. This particular curve would benefit from a higher data point collection rate to remove the stepping effect of the data and improve overall accuracy.

$$S = \frac{m_{\text{standard}} \Delta H_{\text{standard}}}{\int_{t_1}^{t_2} \Delta T(t) dt}$$

S...citlivost na
uvolnění latentní teplo

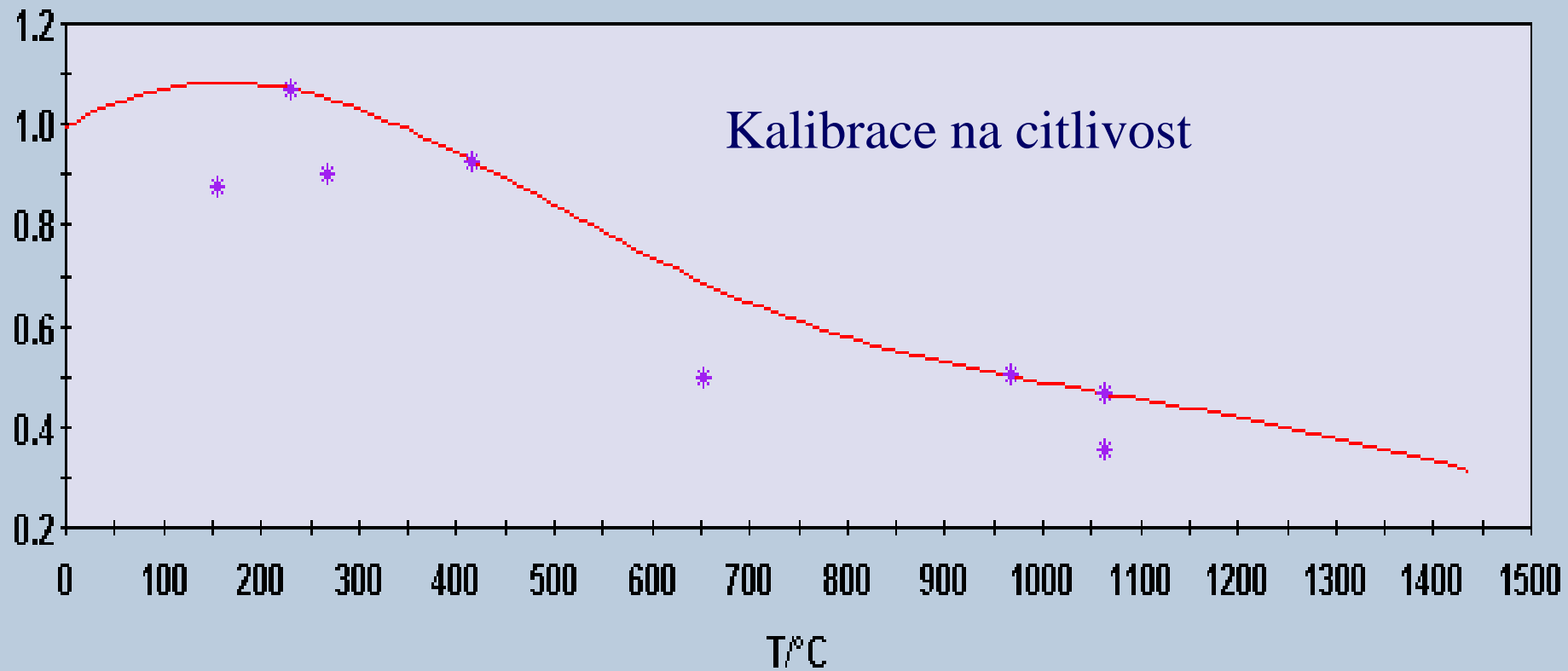
Výpočet pro vzorek:

$$m_{\text{sample}} \Delta H_{\text{sample}} = S \int_{t_1}^{t_2} \Delta T(t) dt$$

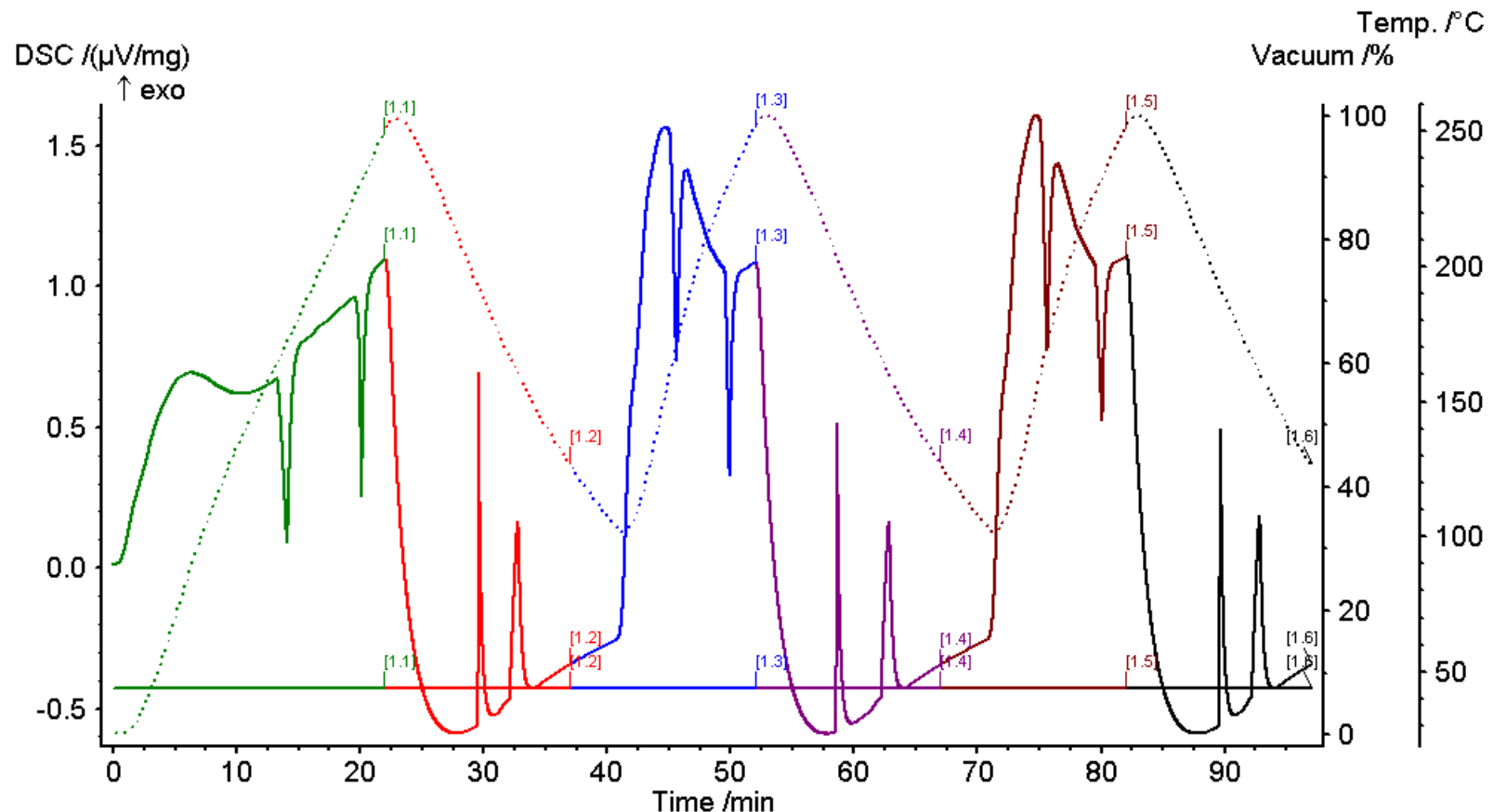
=S.(plocha
a peaku)

Citlivostní závislost

Sensitivity $\mu\text{V}/\text{mW}$

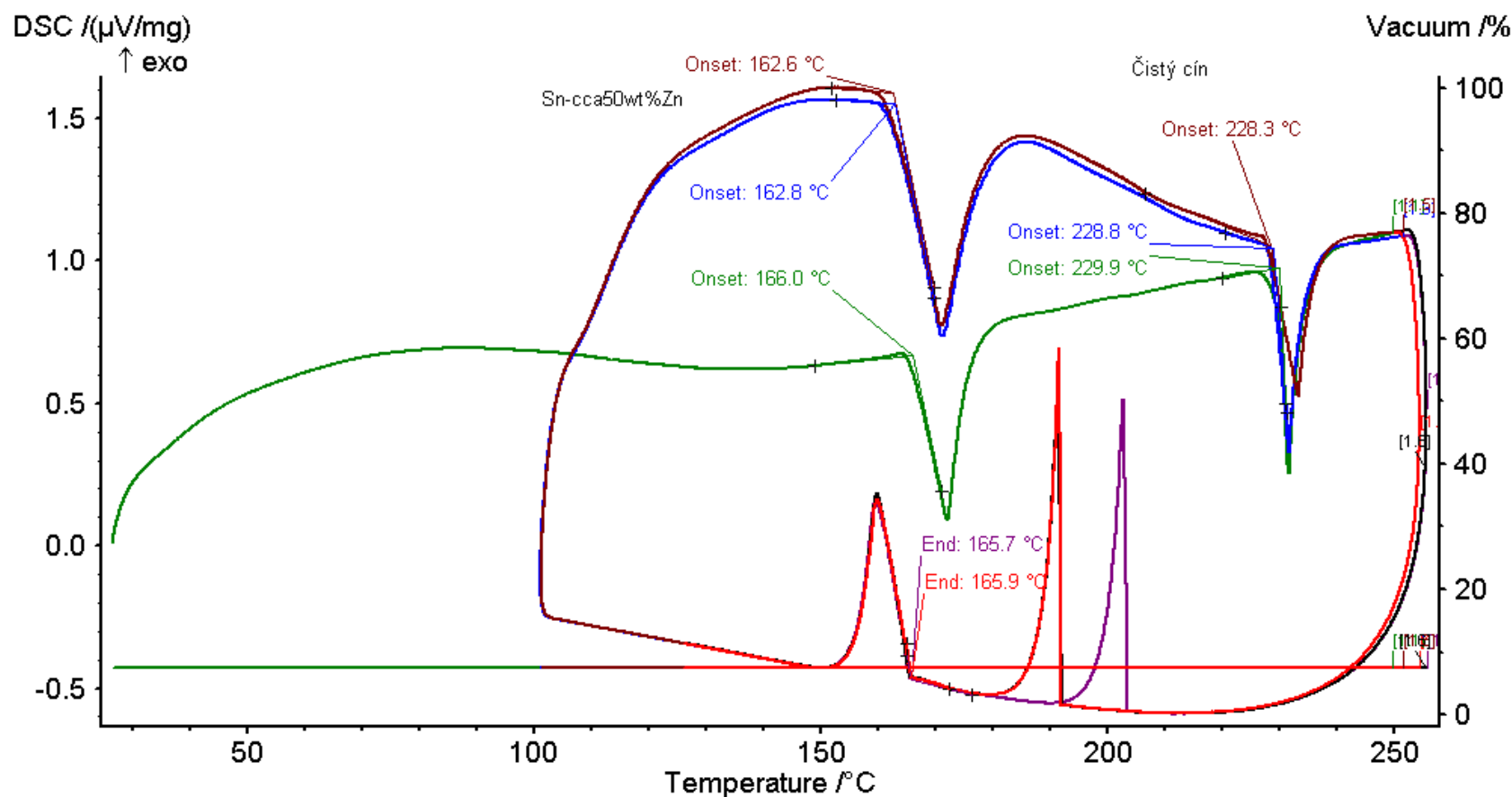


hfDSC měření vzorku



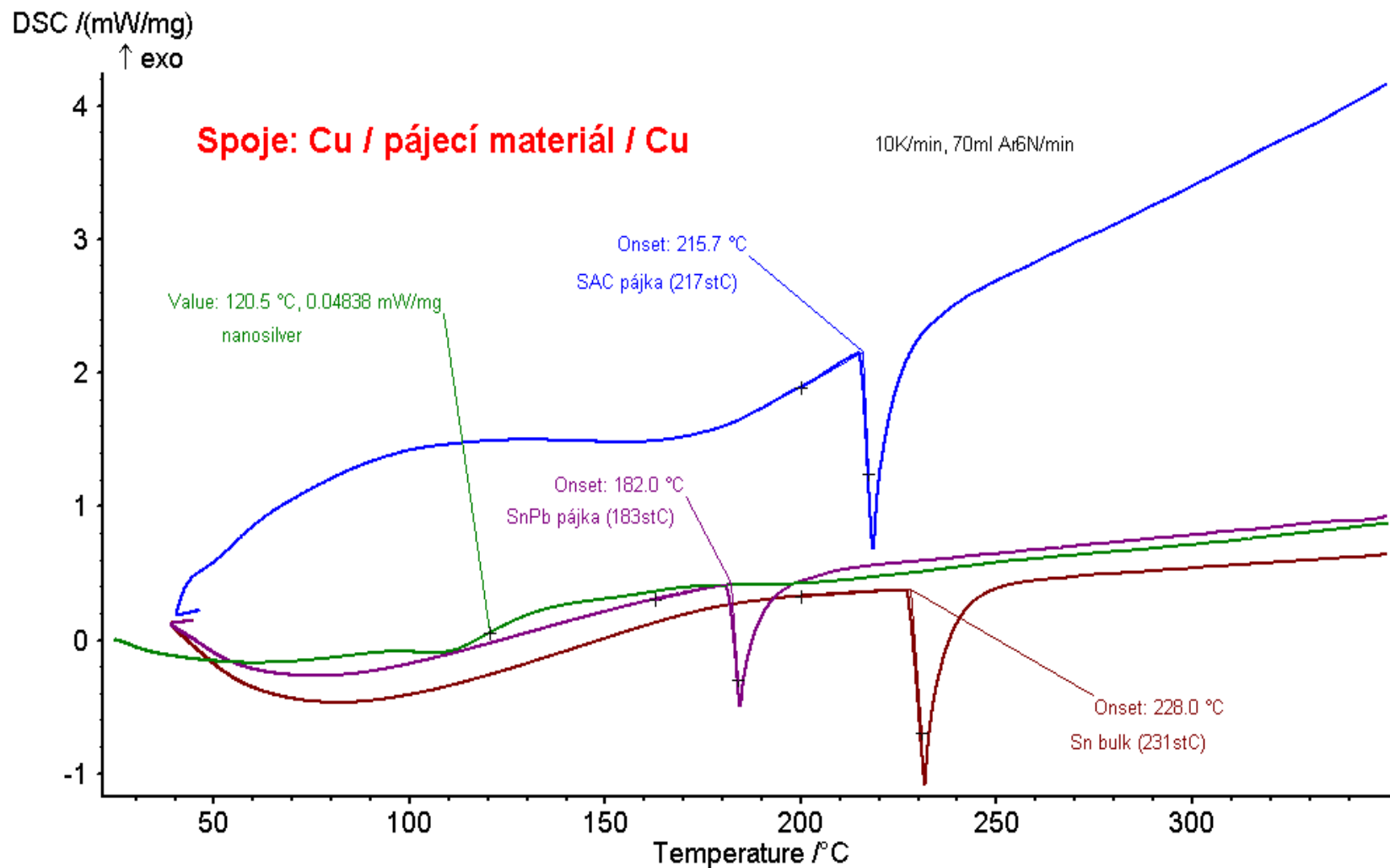
Slitina SnZnSb závislost hfDSC signálu na čase.

Teplotní závislost



Slitina SnZnSb závislost hfDSC signálu na teplotě. 1.2.3 cyklus (run). Reprodukovatelnost při ohřevu, podchlazení při chlazení. (10Kmin, 70mlAr6Nmin).

Tepelné efekty při vzniku spojů mezi Cu substráty



Závislost DSC na rychlosti ohřevu

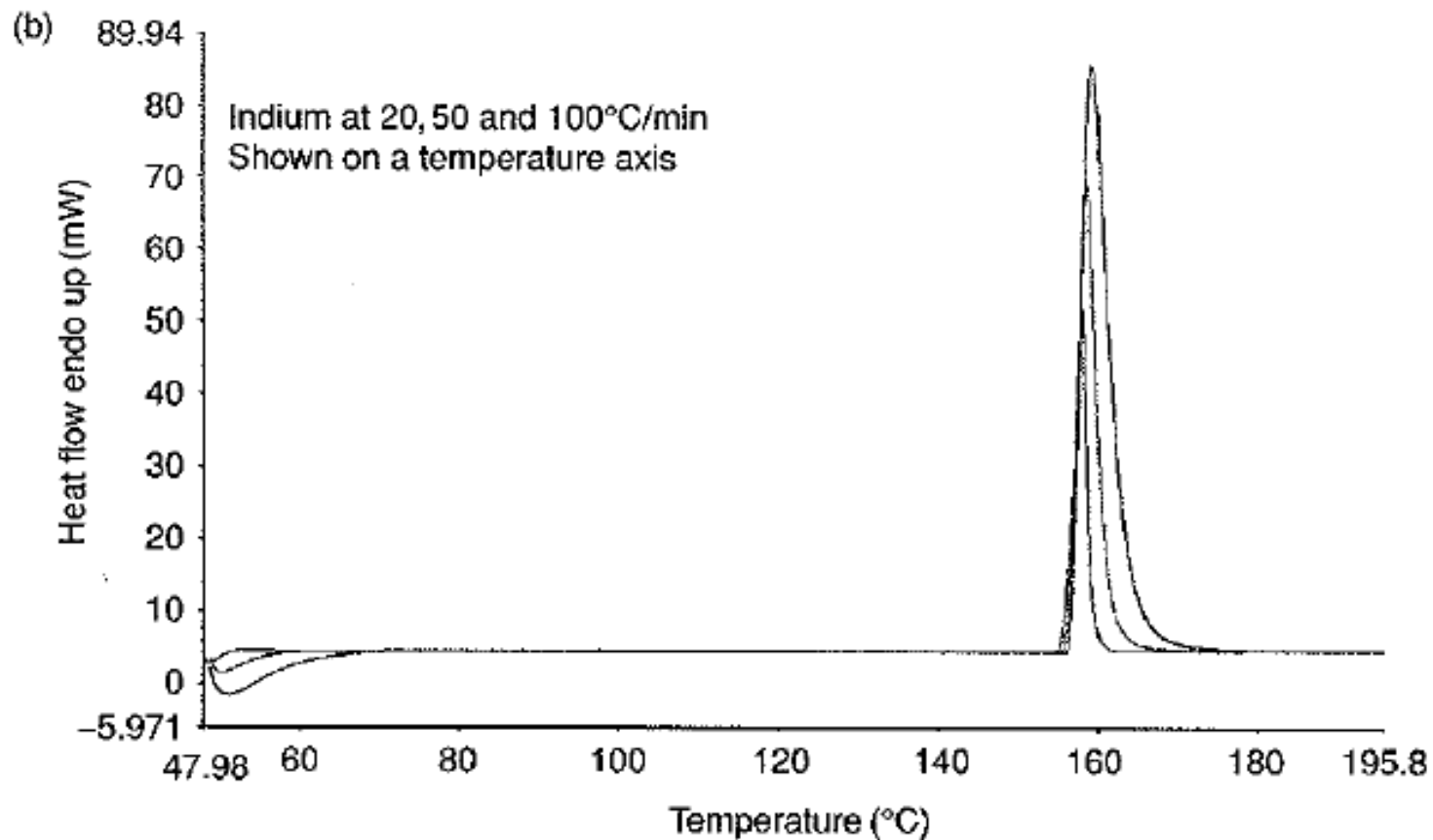
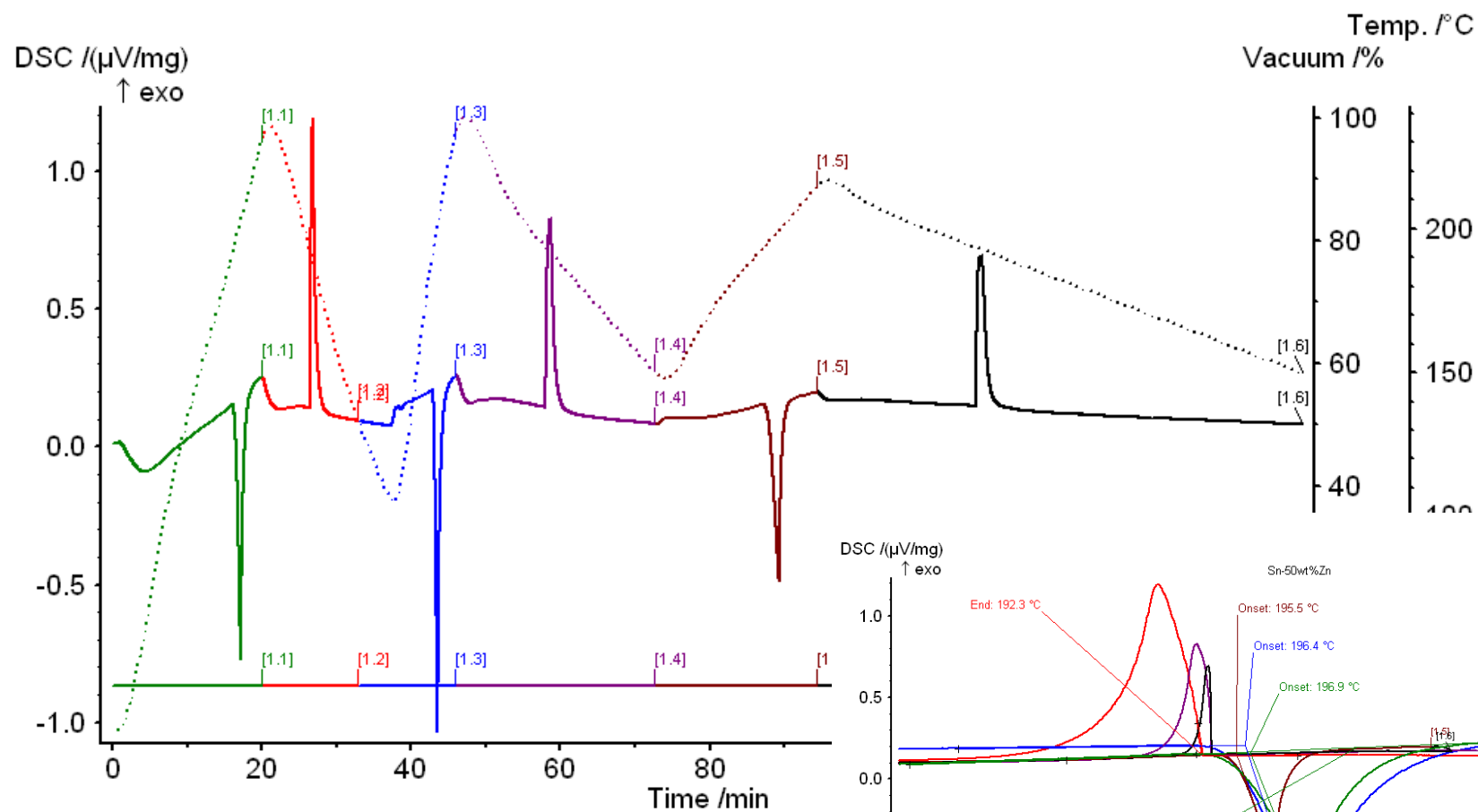
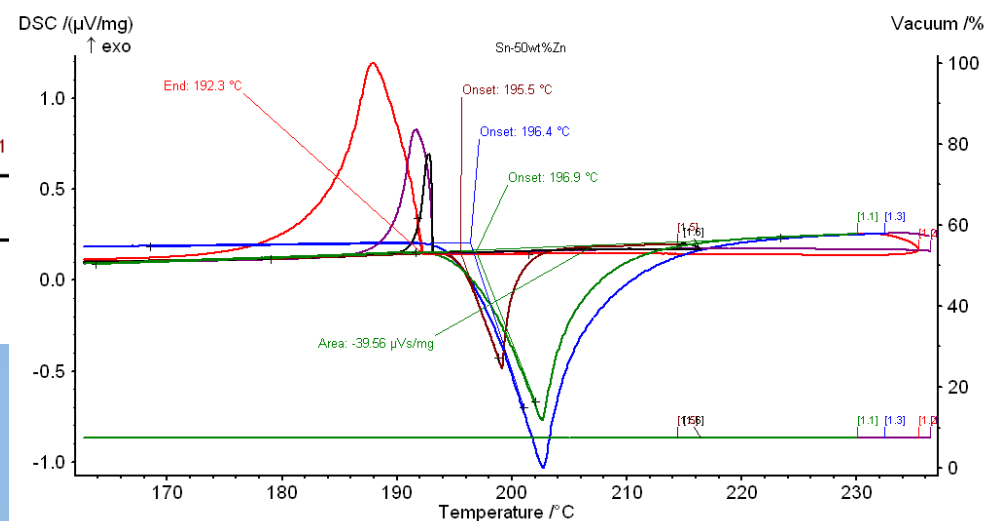


Figure 1.3 The effect of increasing scan rate on indium. Part (a) (upper curve) is shown with the x-axis in time; part (b) (lower curve) is shown with the x-axis in temperature. The same energy flows faster in a

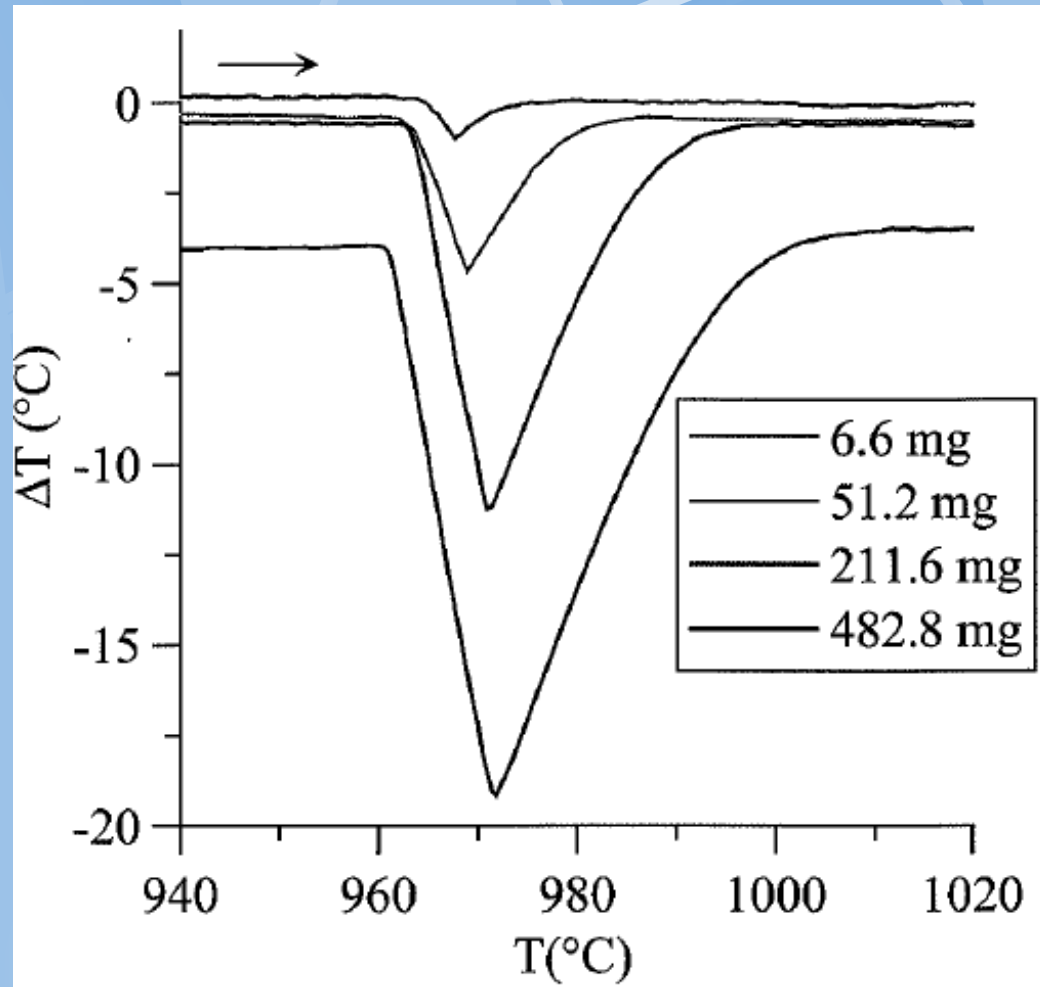
Závislost na rychlosti ohřevu



Sn-50Zn (v argonu)



Závislost na hmotnosti vzorku

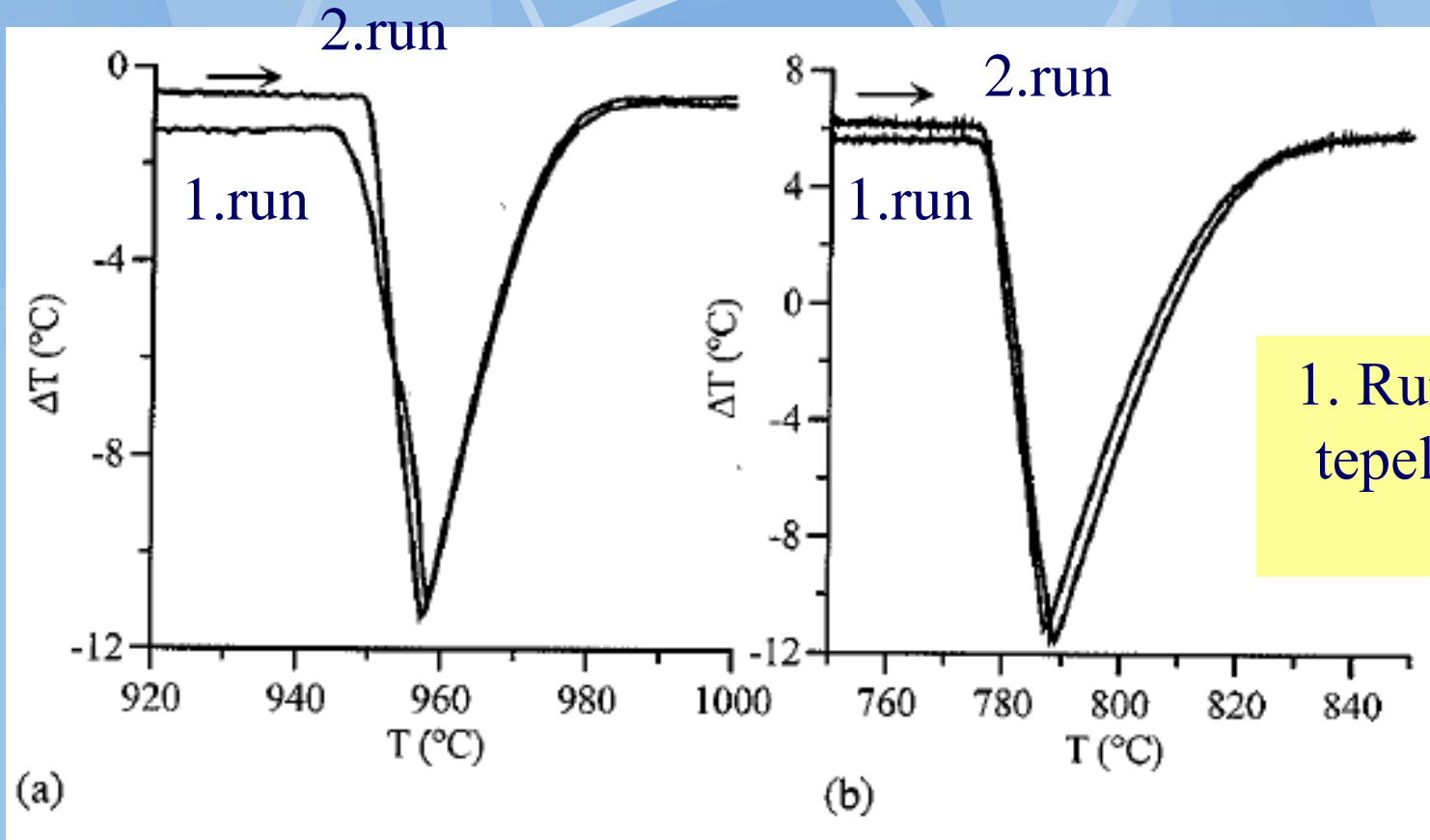


Práškové vzorky
vykazují větší
kontaminaci.

Nejvhodnější je
jeden kousek s
rovnou hranou
(dobrý tepelný
kontakt).

Figure 2.3. Effect of sample mass on DTA signal for pure Ag. The reference mass was held constant. Heating rate is 10 K/min.

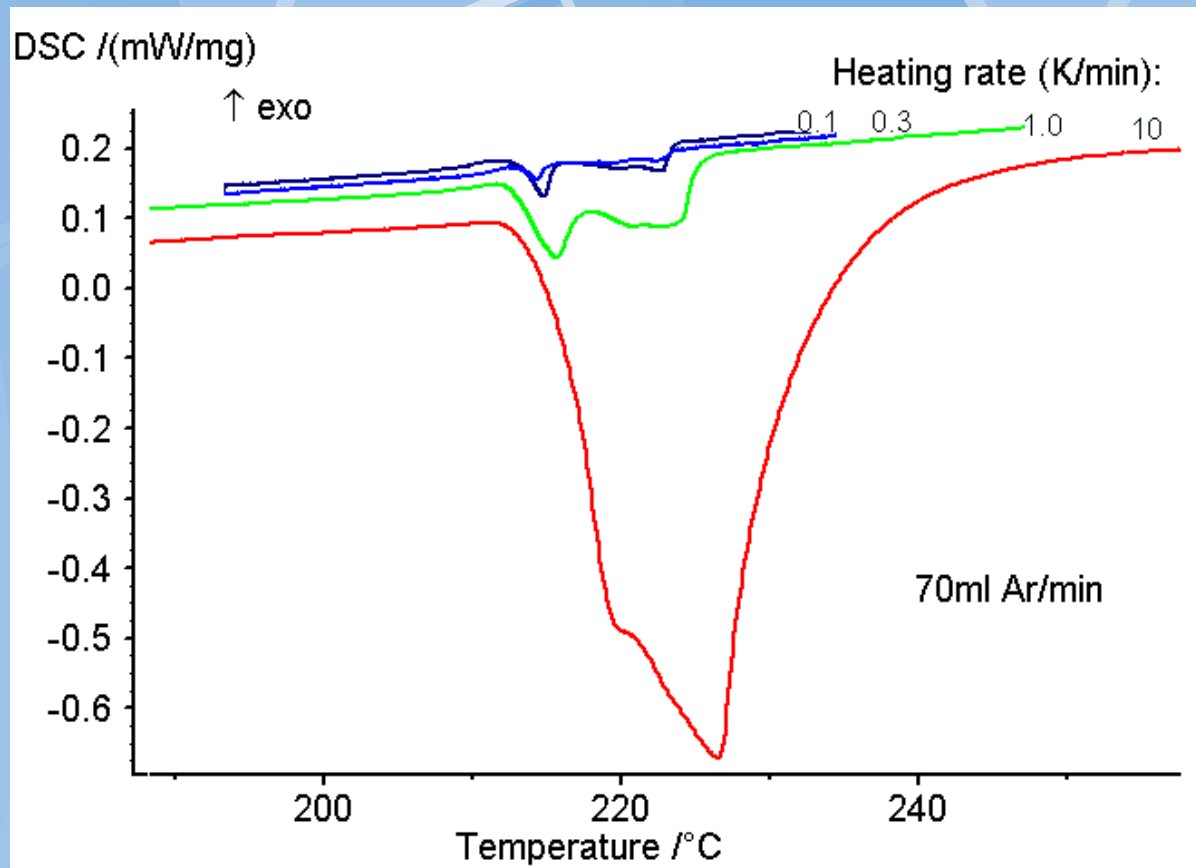
Závislost na pořadí ohřevu



1. Run...vytvoření
tepelně vodivých
spojů

Figure 2.4. DTA signal for melting of (a) pure Ag (211.6 mg; 10 K/min) and (b) Ag-Cu eutectic (231.3 mg; 15 K/min) comparing first melt (red) and second melt (blue). Note the offset of the baselines, the more rounded onsets, and the lack of linearity of the down-slopes for the first melt compared to the second.

Separace signálu hfDSC



Vzorek
AgSnBi:
70mlAr6Nmin

Posuv k
termodynamické
rovnováze

Fázové transformace 1. řádu

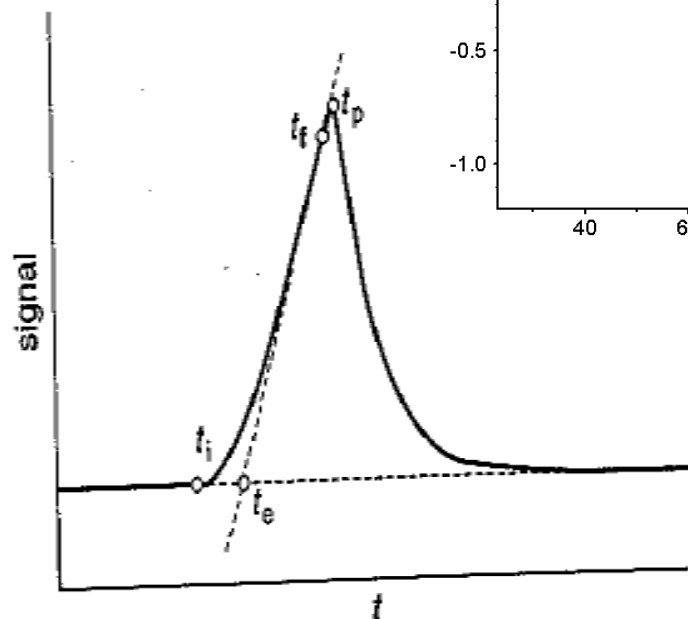
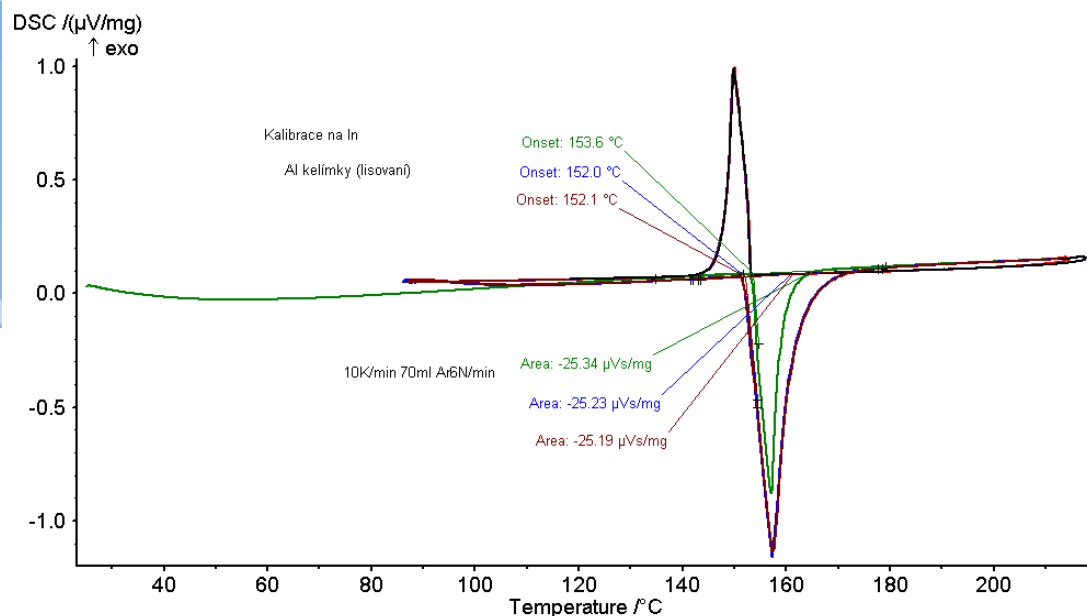


Figure 2 Typical trace plotted against time for a first-order phase transition. t_i , signal starts to deviate from the baseline; t_e , extrapolated onset; t_f , inflection point where the slope of the trace is maximum; t_p , peak maximum



**hfDSC-
Indium
STA409**

FT 2. řádu

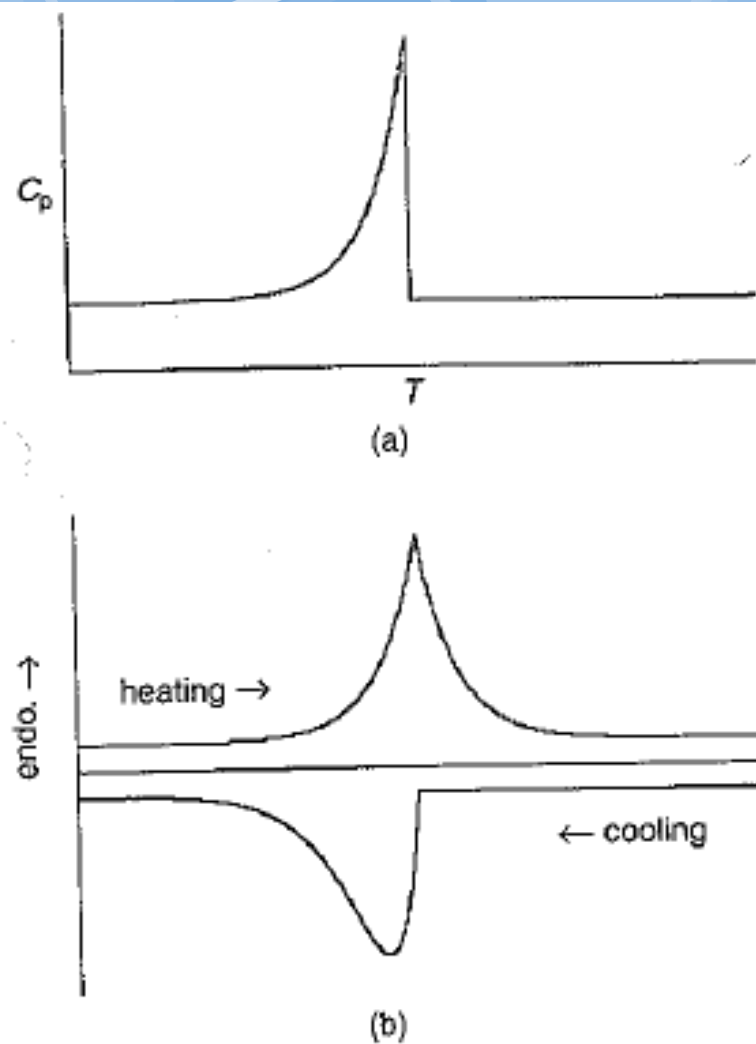


Figure 3 Heat capacity (a) and idealized trace (b) in classical DTA and power-compensated DSC for a second-order phase transition

Ideální a reálné chování signálu pcDSC

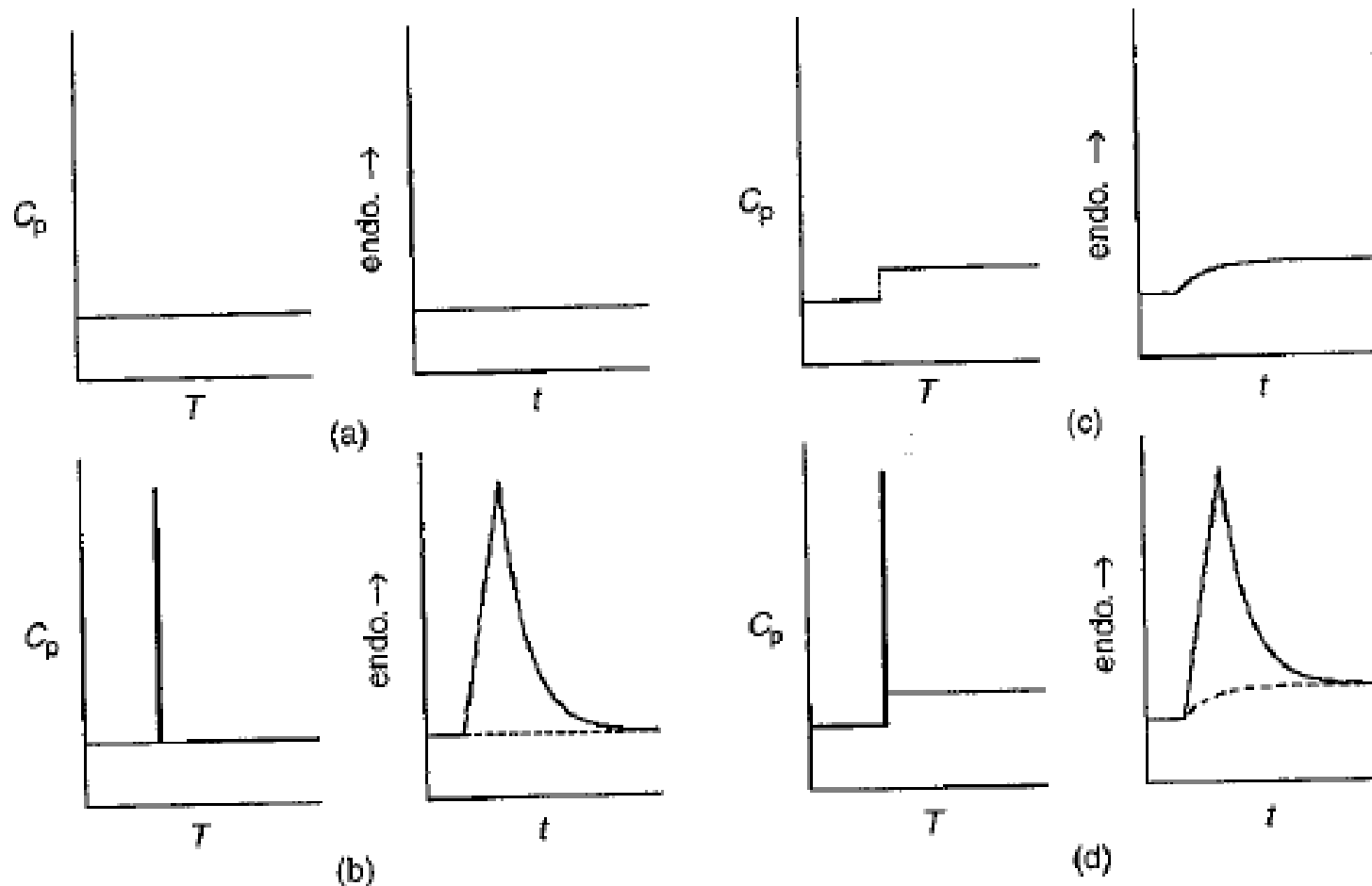
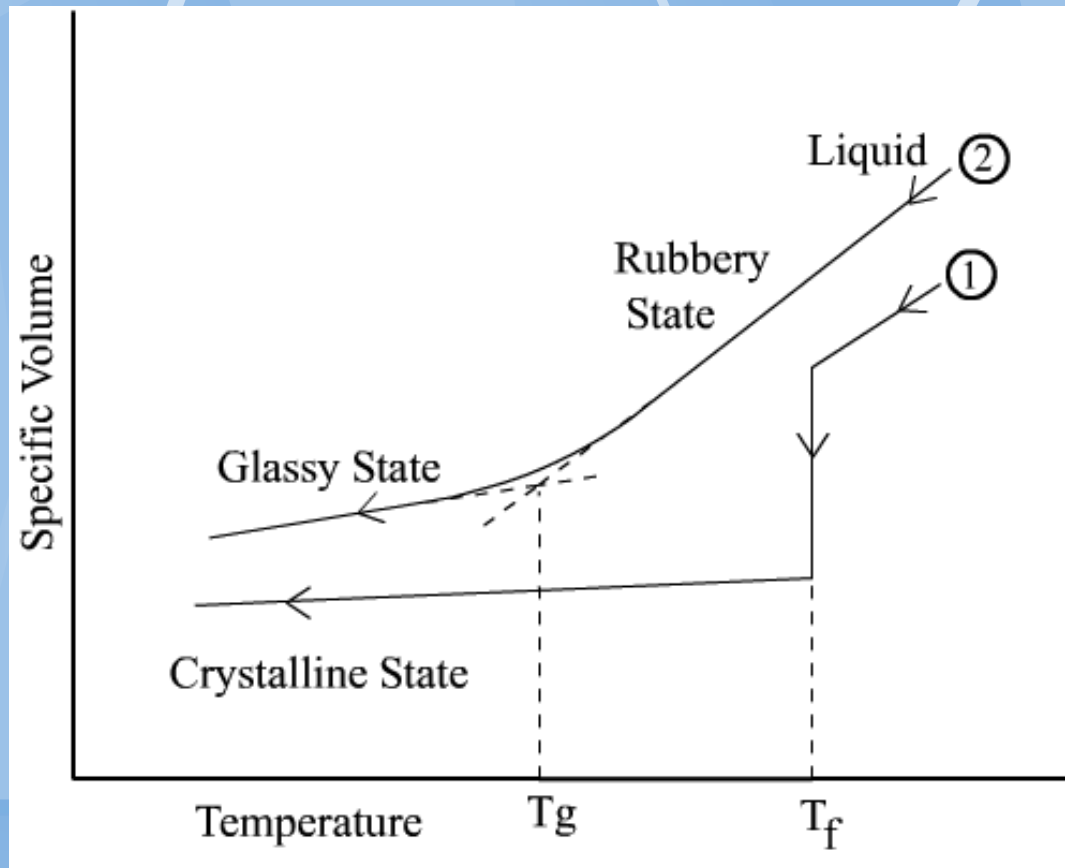


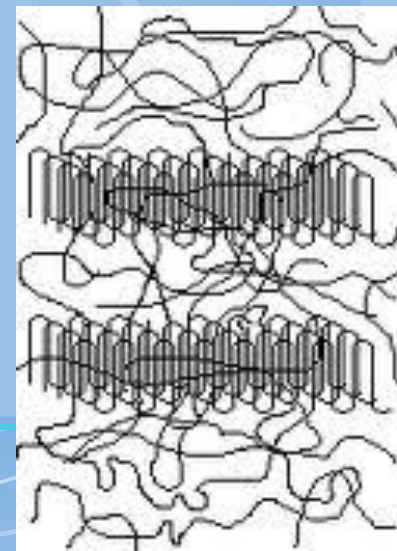
Figure 4 Principle of drawing baseline to separate a thermal event. Thermal behaviors (left) and corresponding ideal traces in power-compensated DSC (right)

Glass transition (skelný přechod) T_g

Skelný přechod je reverzibilní přeměna, kdy amorfní látka (často polymer) viskózního nebo „gumovitého“ charakteru přechází do stavu tuhého a relativně křehkého.



Teplota skelného přechodu T_g je teplota, která reprezentuje oblast kdy dochází k skelnému přechodu



Důsledky T_g

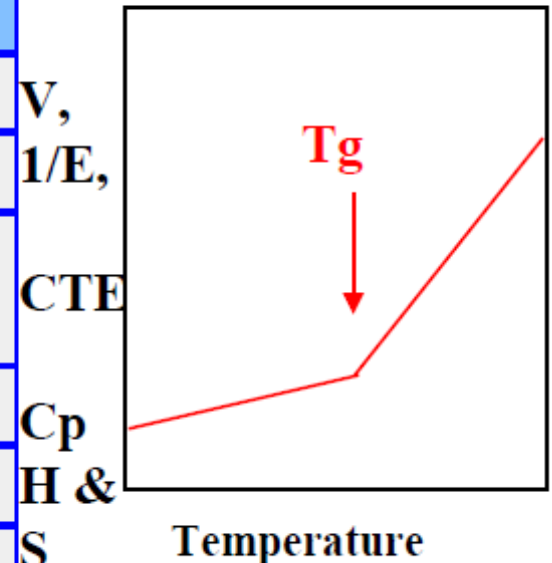


PLA below T_g

PLA above T_g

Změna vlastnosti při T_g

Physical property	Response on heating through T_g
Specific Volume	Increases
Modulus	Decreases
Coefficient of thermal expansion	Increases
Specific Heat	Increases
Enthalpy	Increases
Entropy	Increases

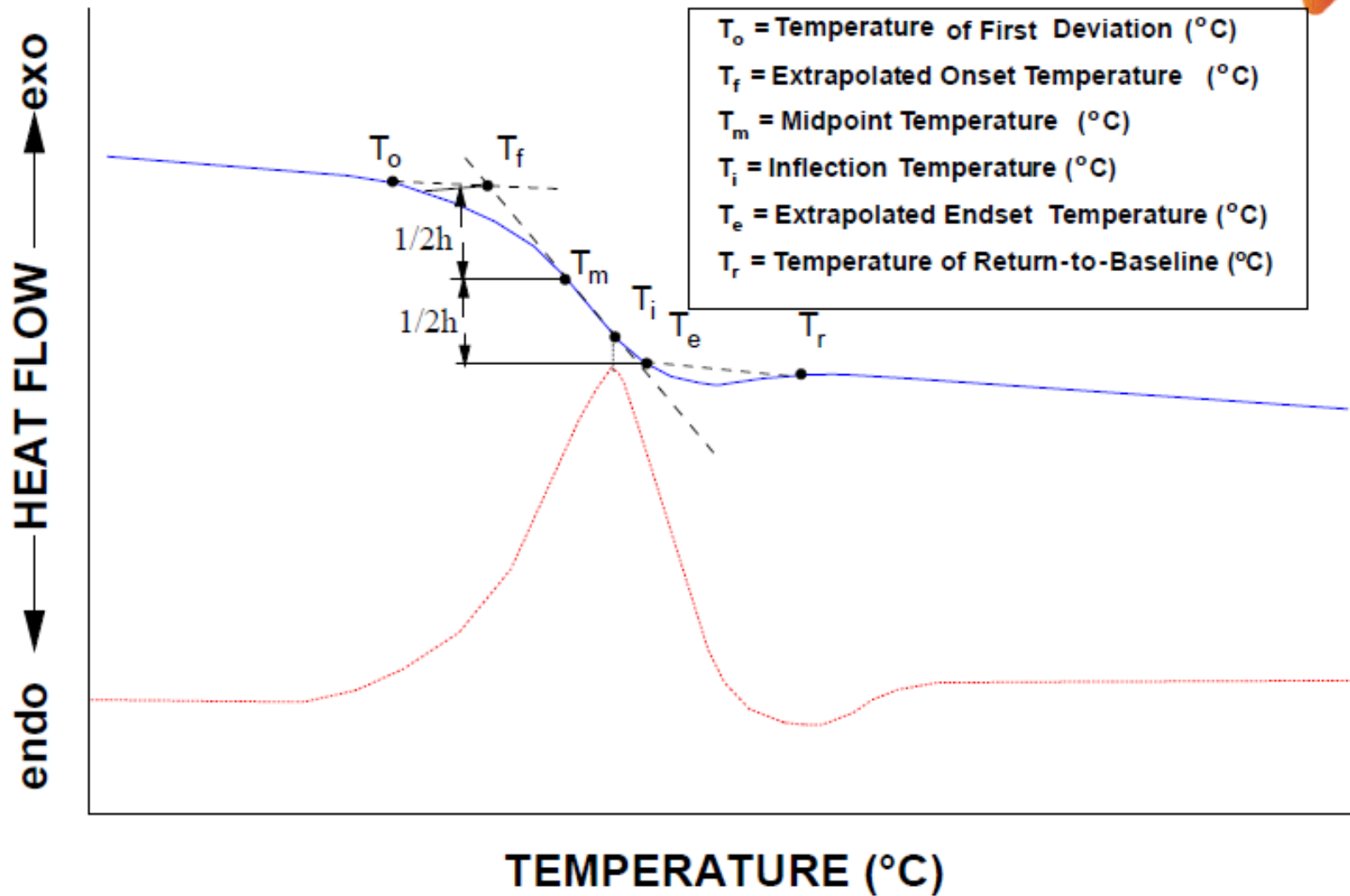


T_g závisí na:

Heating Rate
Heating & Cooling
Aging
Molecular Weight
Plasticizer
Filler

Crystalline Content
Copolymers
Side Chains
Polymer Backbone
Hydrogen Bonding

Vyhodnocení T_g z DSC signálu



Glass Transition Temperature for Selected Polymers

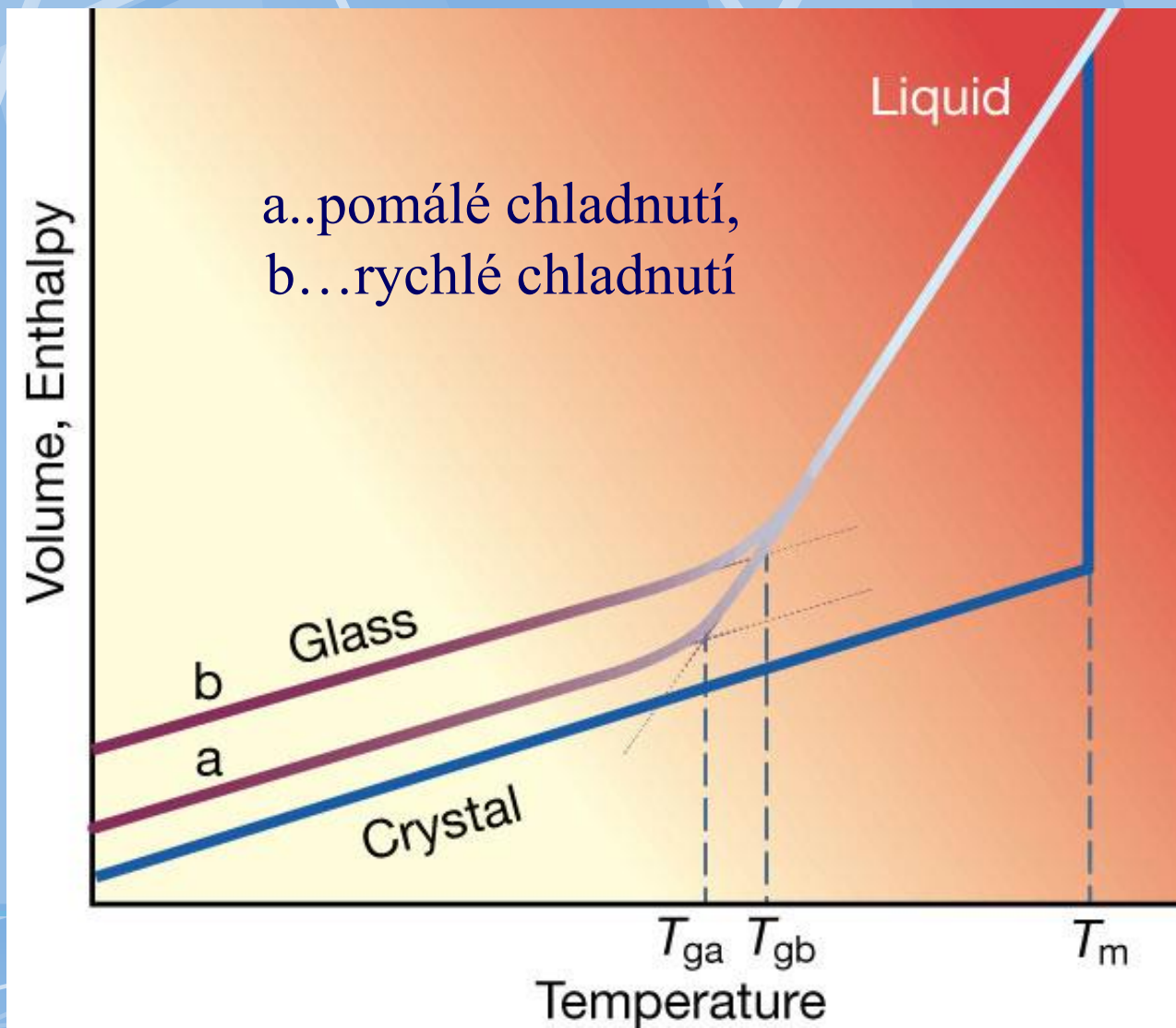
Organic Polymer	T_g (°C)
Polyacenaphthalene	264
Polyvinyl pyrrolidone	175
Poly- <i>o</i> -vinyl benzyl alcohol	160
Poly- <i>p</i> -vinyl benzyl alcohol	140
Polymethacrylonitrile	120
Polyacrylic acid	106
Polymethyl methacrylate	105
Polyvinyl formal	105
Polystyrene	100
Polyacrylonitrile	96
Polyvinyl chloride	87
Polyvinyl alcohol	85
Polyvinyl acetal	82
Polyvinyl propional	72
Polyethylene terephthalate	69
Polyvinyl isobutyral	56
Polycaprolactam (nylon 6)	50
Polyhexamethylene adipamide (nylon 6,6)	50
Polyvinyl butyral	49
Polychlorotrifluoroethylene	45
Ethyl cellulose	43

Termop
lasty

Organic Polymer	T_g (°C)
Polyhexamethylene sebacamide (nylon 6,10)	40
Polyvinyl acetate	29
Polyperfluoropropylene	11
Polymethyl acrylate	9
Polyvinylidene chloride	-17
Polyvinyl fluoride	-20
Poly-1-butene	-25
Polyvinylidene fluoride	-39
Poly-1-hexene	-50
Polychloroprene	-50
Polyvinyl- <i>n</i> -butyl ether	-52
Polytetramethylene sebacate	-57
Polybutylene oxide	-60
Polypropylene oxide	-60
Poly-1-octene	-65
Polyethylene adipate	-70
Polyisobutylene	-70
Natural rubber	-72
Polyisoprene	-73
Polydimethyl siloxane	-85
Polydimethyl siloxane	-123

Gumy

Závislost T_g na rychlosti změny teploty



Určování fázových diagramů pomocí DSC

Jednoduché bináry

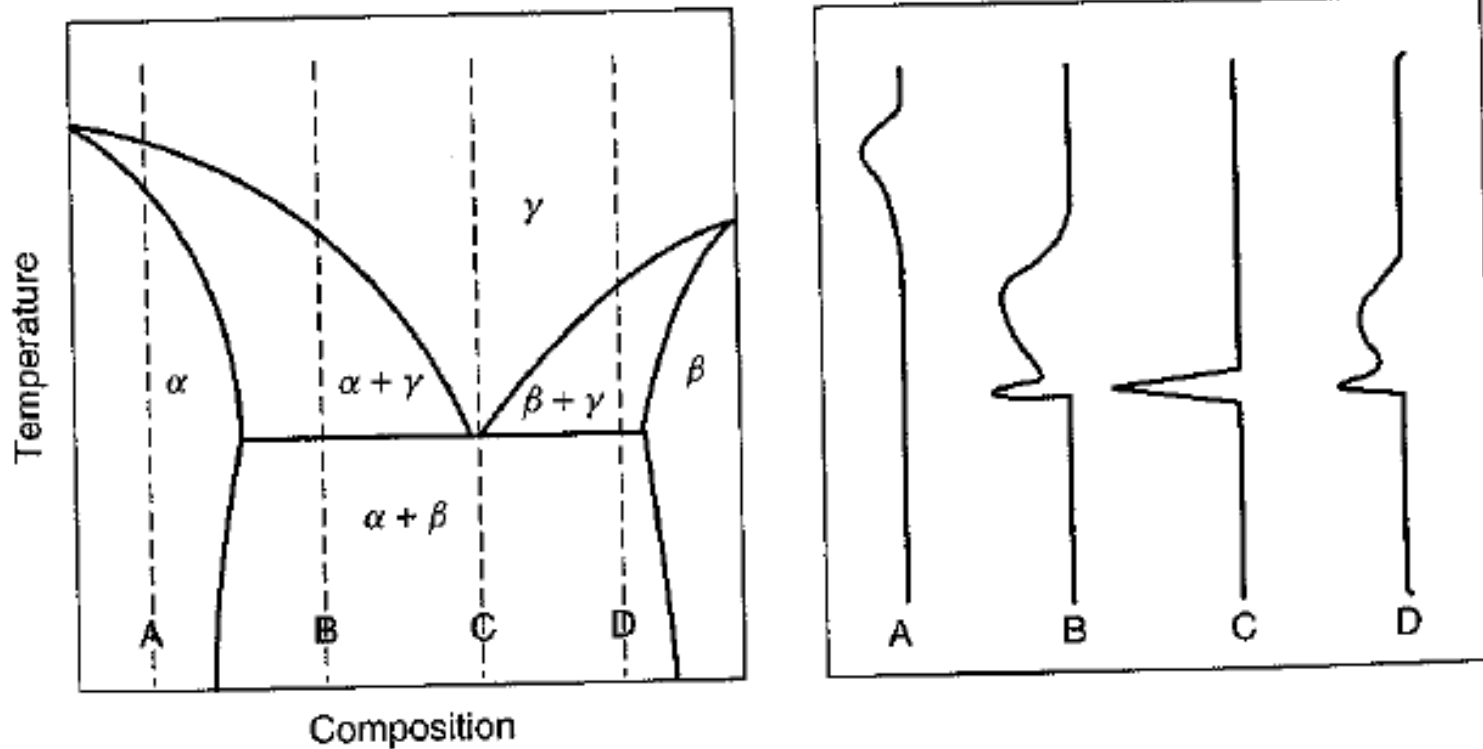


Figure 1 Schematic phase diagram for a binary system with a eutectic phase transition $\alpha + \beta \rightarrow \gamma$, and the corresponding DSC curves for compositions A, B, C and D respectively

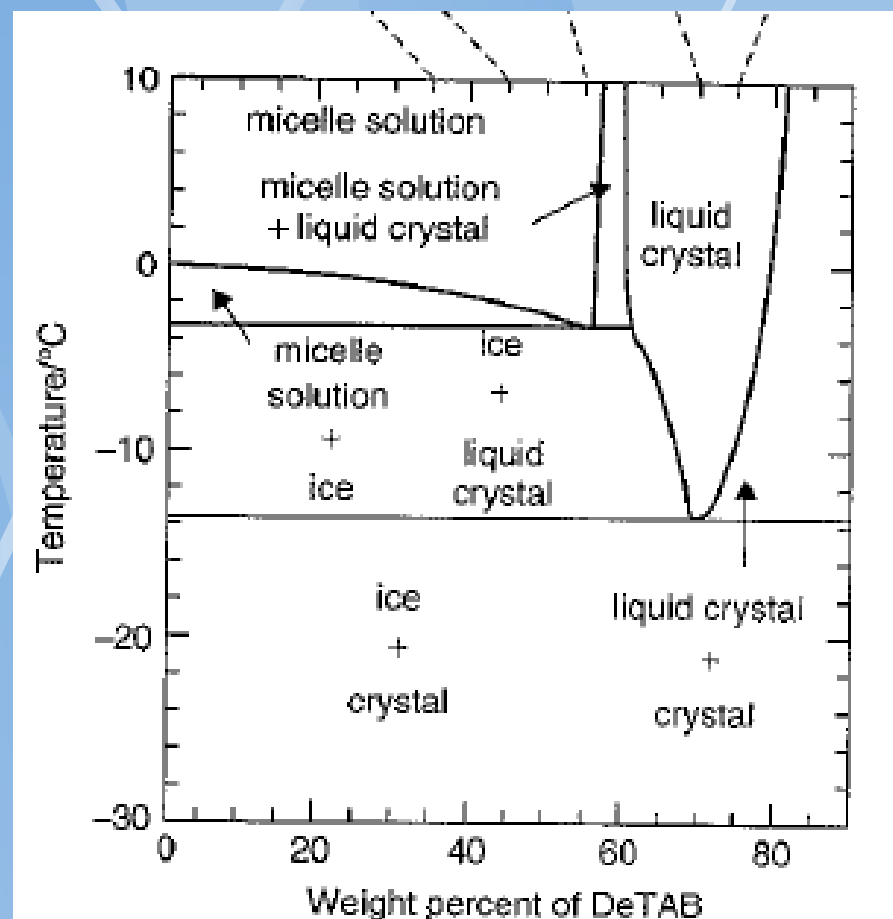
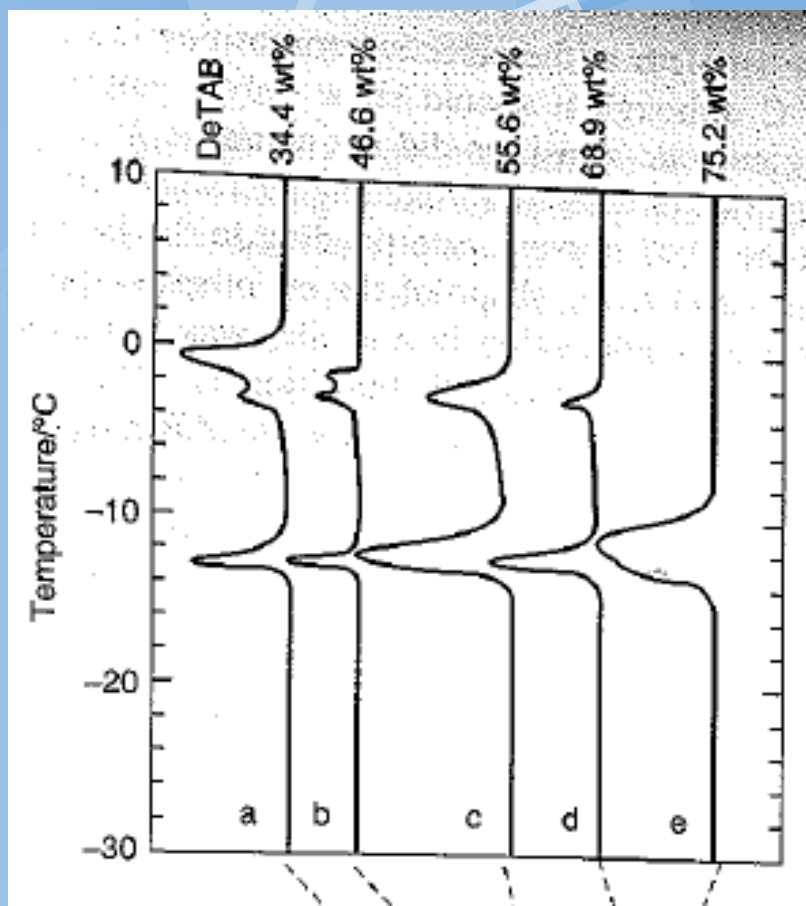
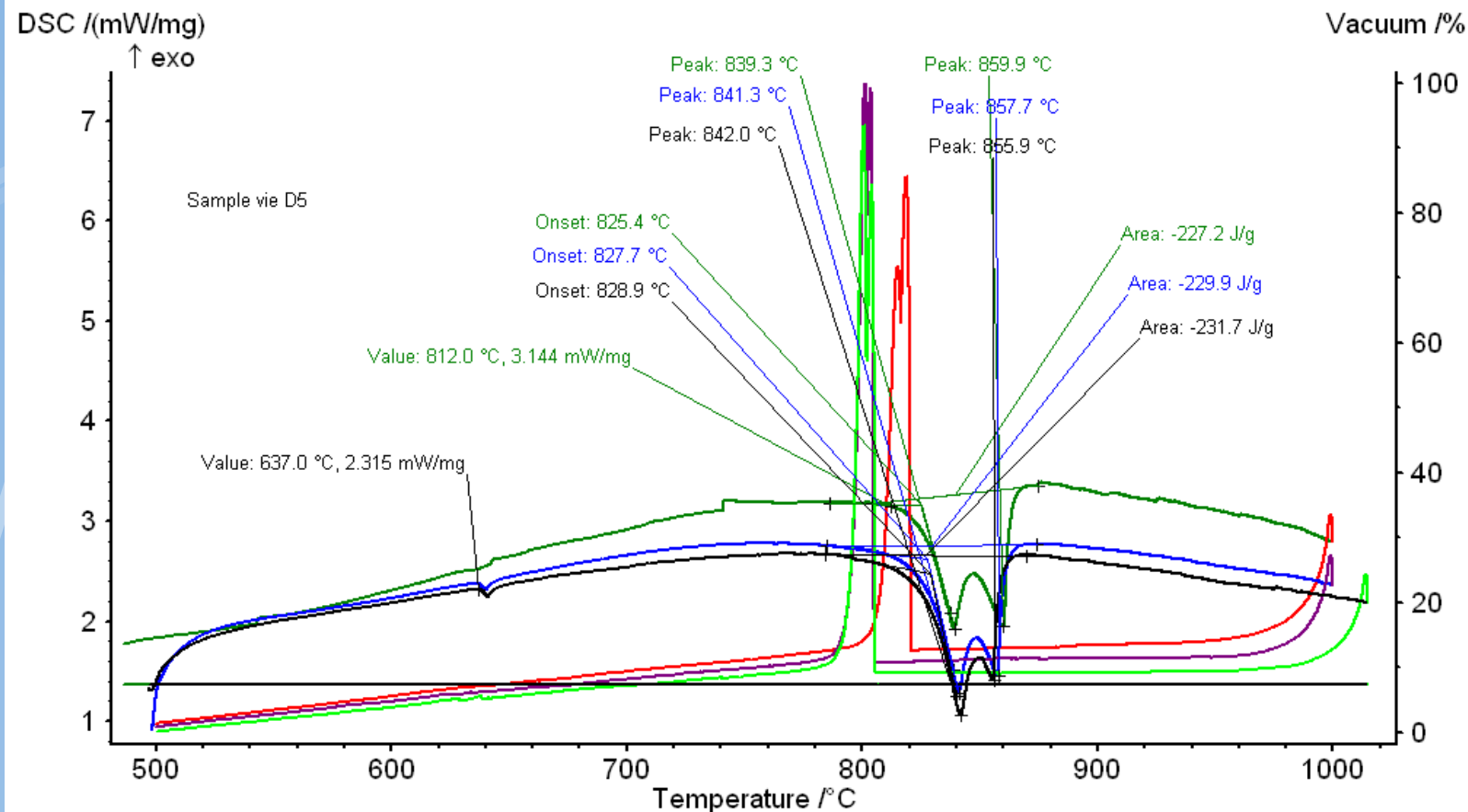


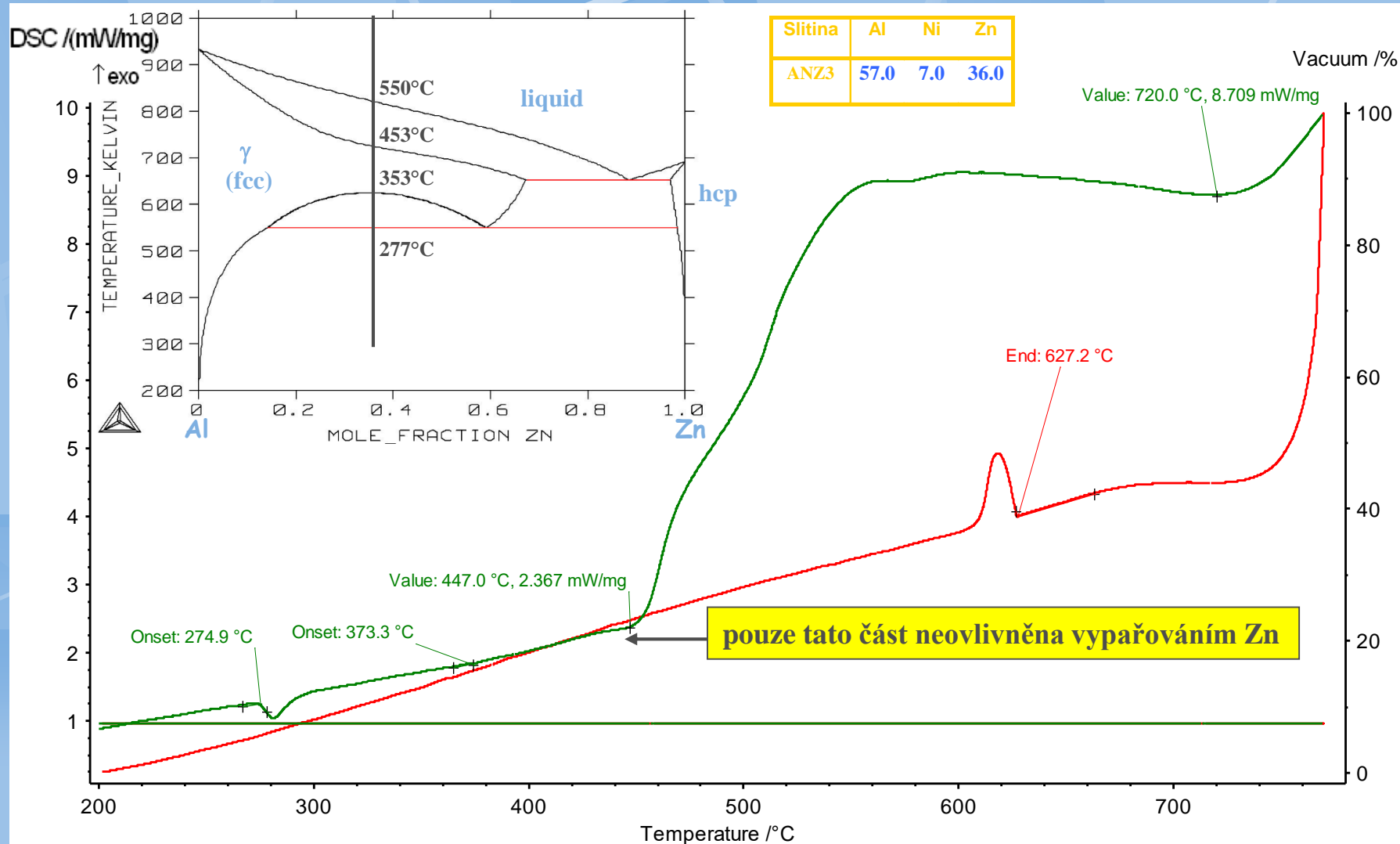
Figure 2 DSC heating curves for DeTAB–water mixtures with various compositions a, b, c, d, and e, and the phase diagram of the DeTAB–water binary system²

FD vícesložkových soustav



Slitina 9.26at%Ag-14,81%Ba-81,48%Ge

DSC měření Al-Ni-Zn



Vyhodnocení DSC křivek pro vzorek ANZ3

Stanovení čistoty

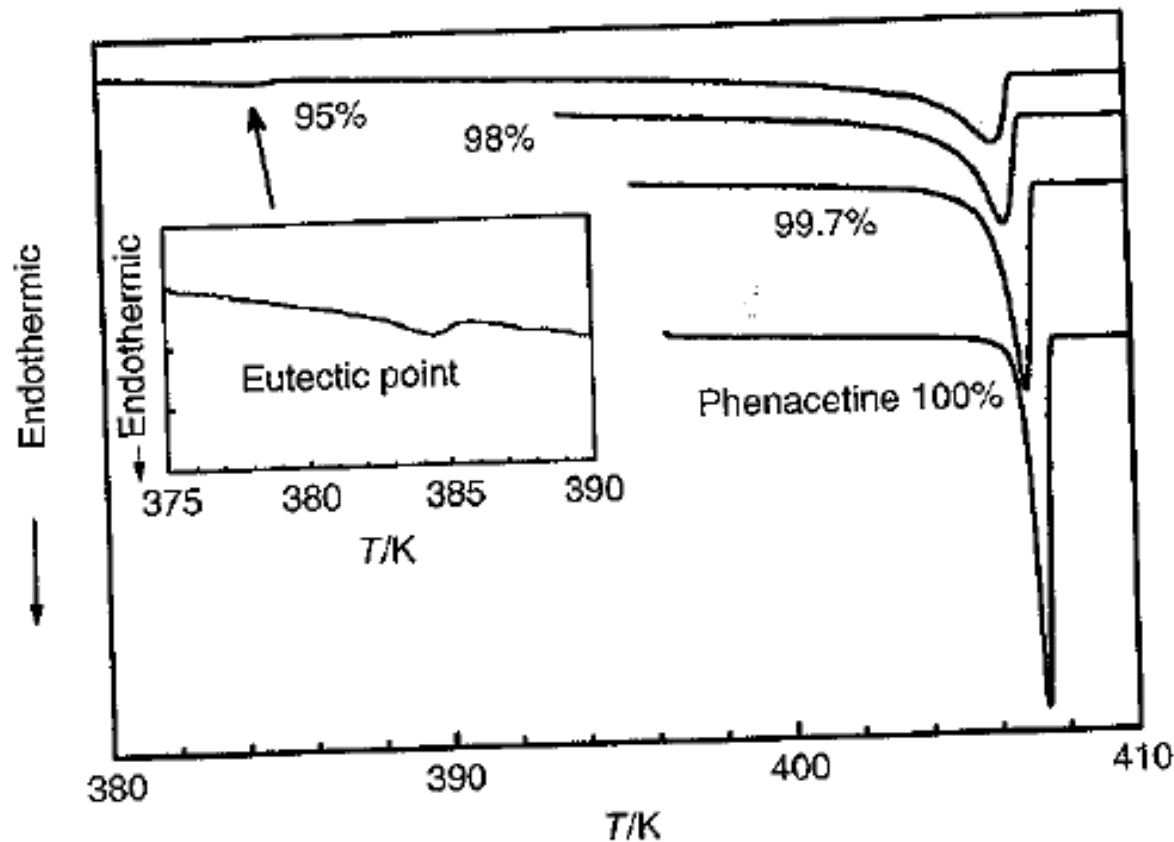
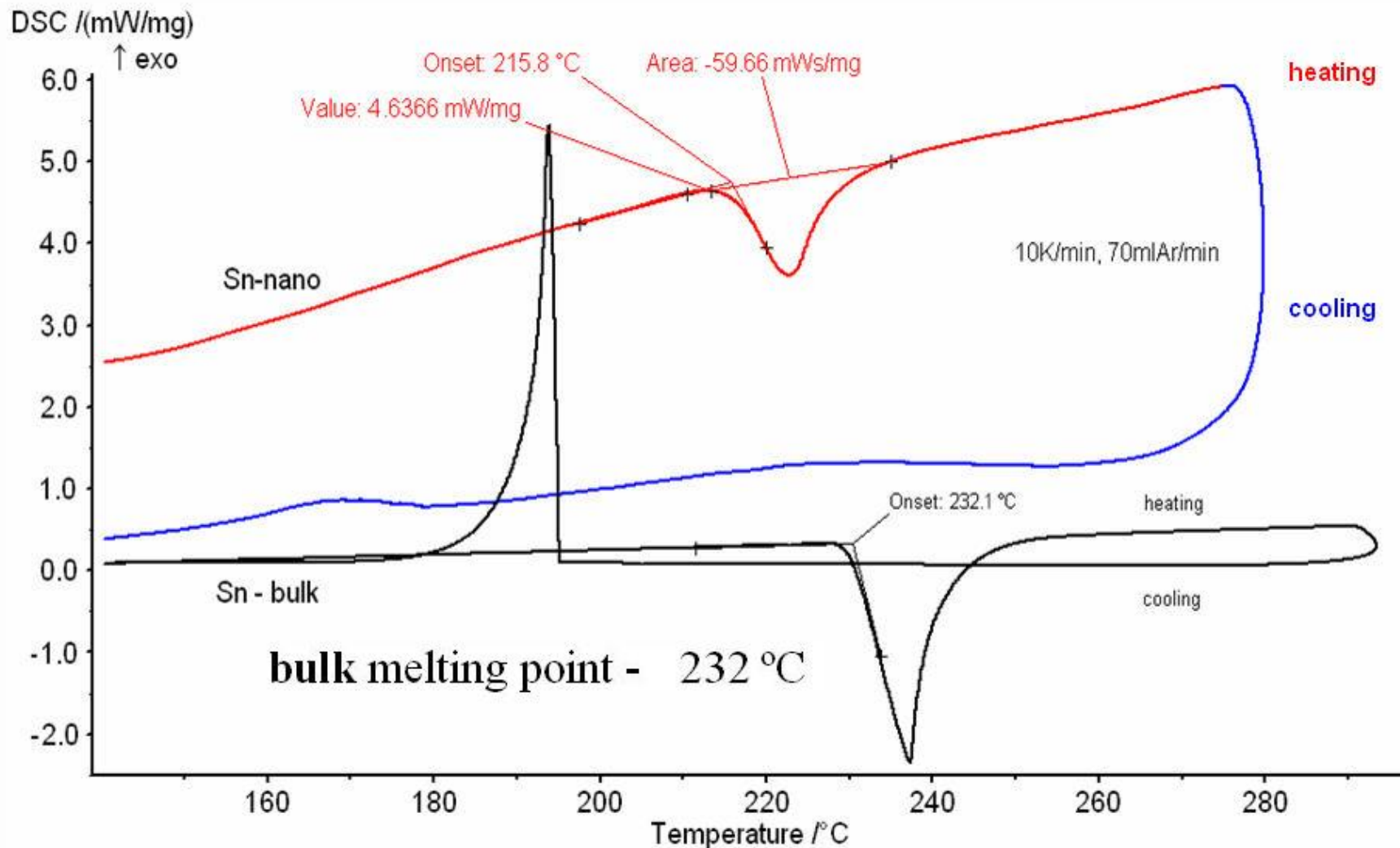


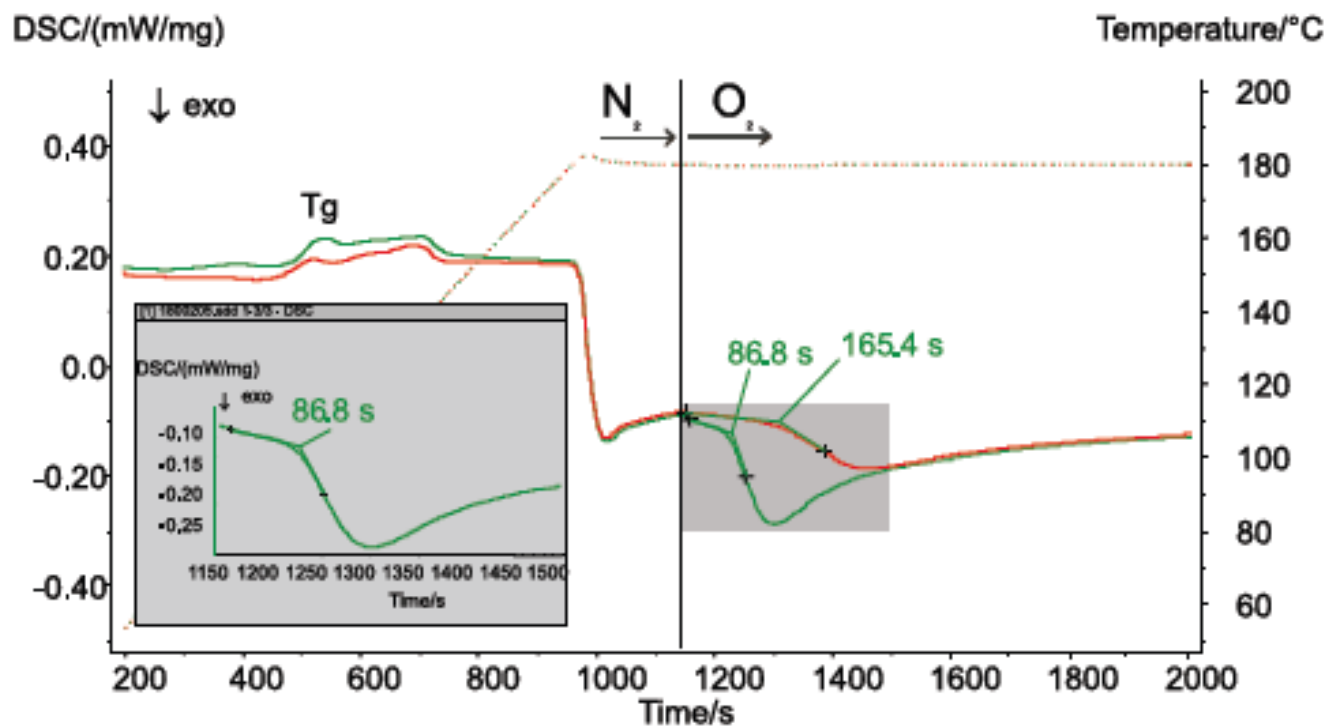
Figure 1 DSC curves of NIST SRM1514 (phenacetines doped with nominal 0.0, 0.7, 2.0 and 5.0 mol % of *p*-aminobenzoic acid)

Sn nano



Melting and crystallisation. Bulk tin (black line). Nanoscale tin (red and blue lines) revealing melting point depression.

Výměna plynu



Isotermní krystalizace

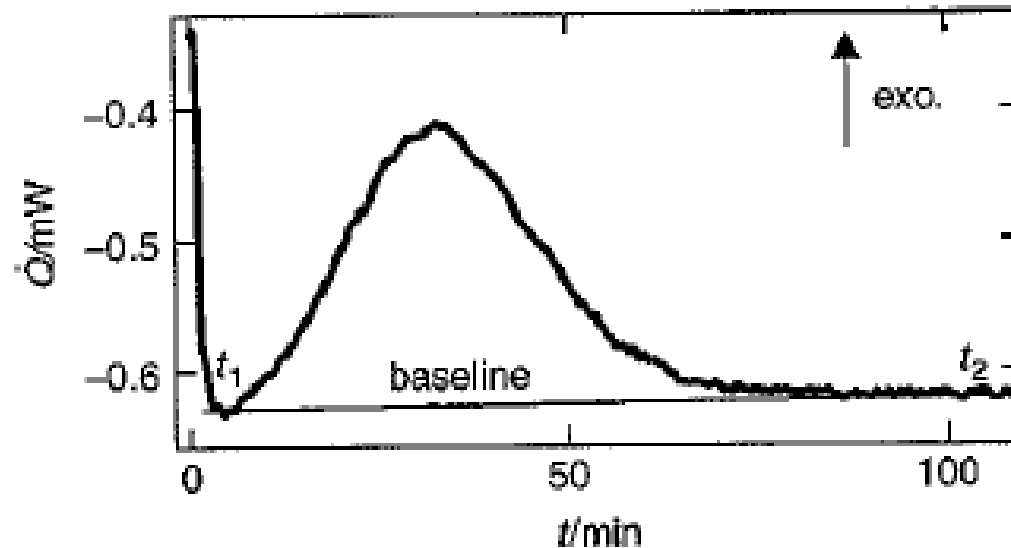


Figure 1 Thermogram of isothermal crystallization of poly(vinylidene fluoride)

Model: Avrami

Adsorbce a desorbce H₂ ve slitinách

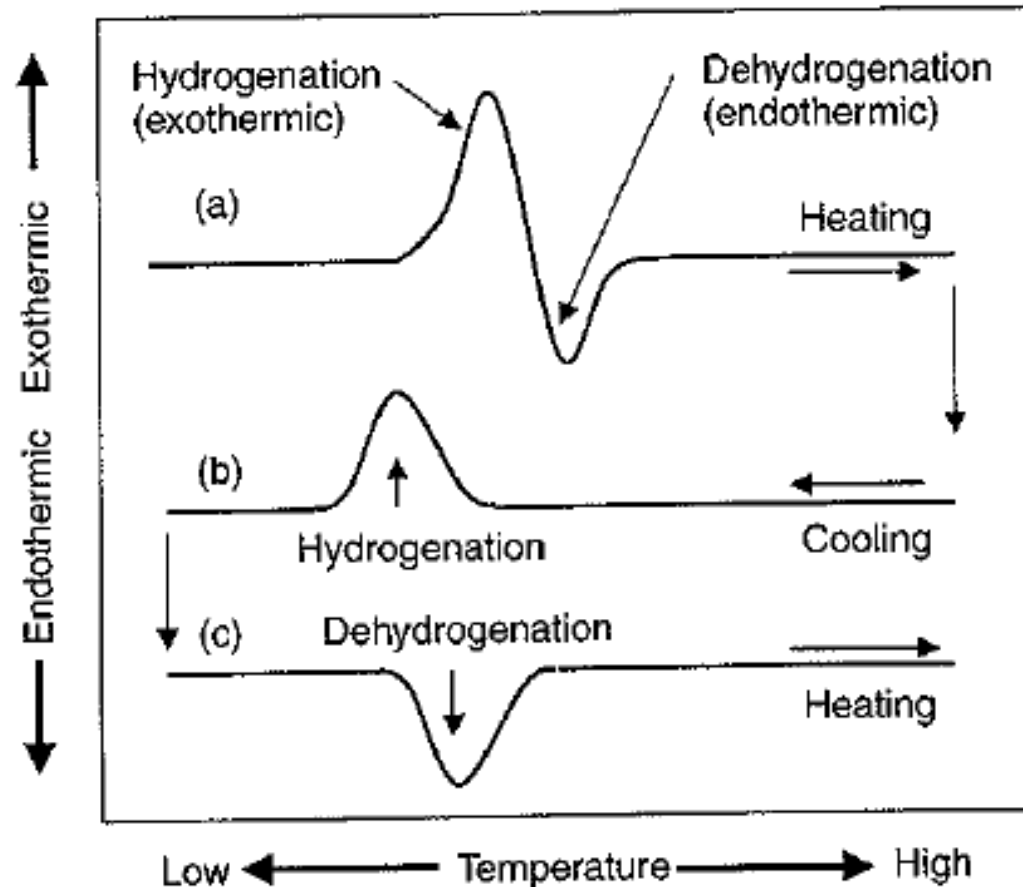
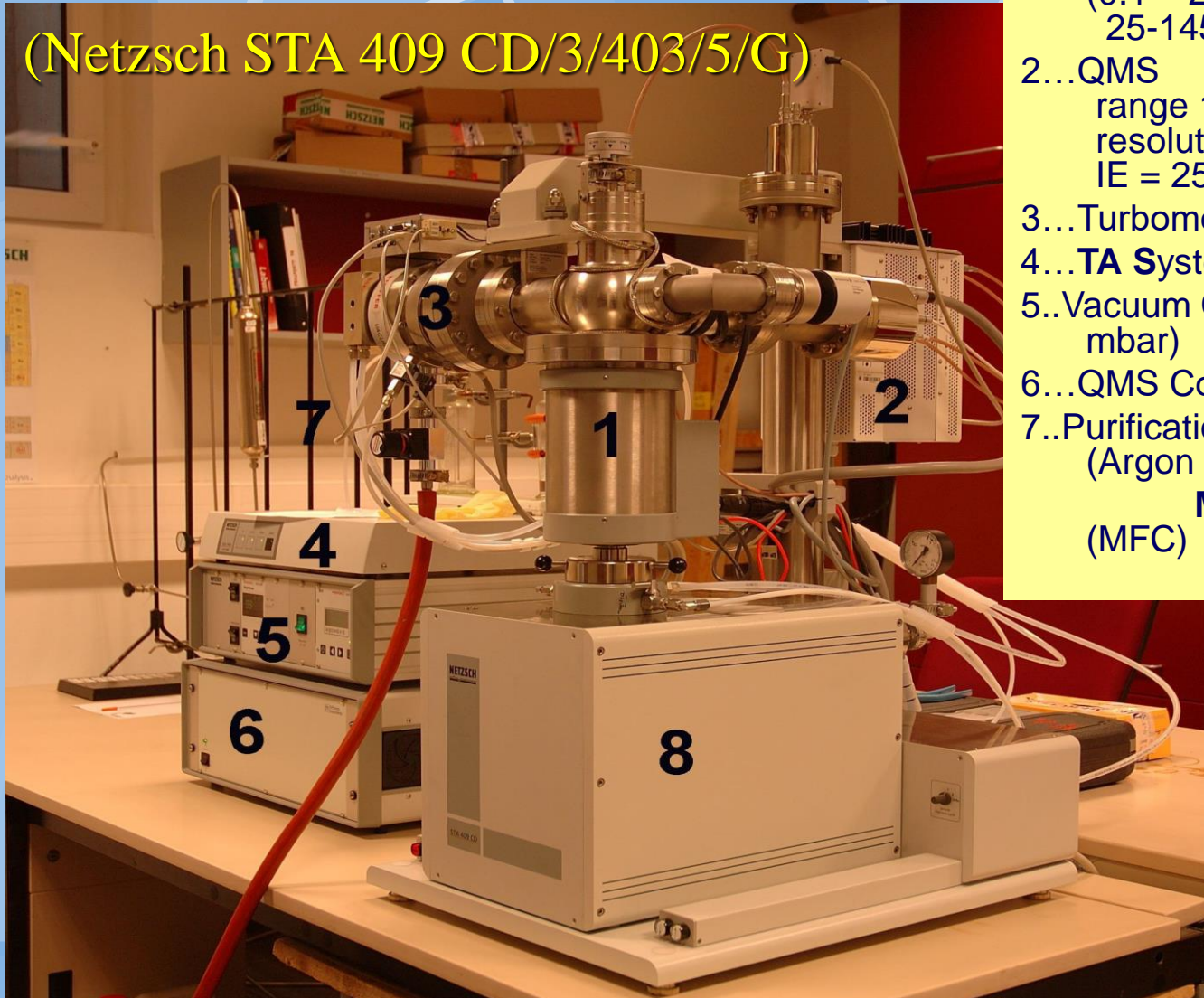


Figure 1 Schematic traces of DTA or DSC of a hydrogen-absorbing alloy under hydrogen atmosphere

Diskuze: STA 409

(Netzsch STA 409 CD/3/403/5/G)



- 1...Furnace
(0.1 – 20 K min⁻¹,
25-1450°C)
- 2...QMS
range 1-512 amu
resolution 0,5amu
IE = 25 -100 eV
- 3...Turbomolecular Pump
- 4...**TA System Controller (TASC)**
- 5..Vacuum Controller, (cca 9·10⁻⁶
mbar)
- 6...QMS Controller
- 7..Purification Column (oxygen)
(Argon 99,999)
Mass Flow Controller (MFC)

Reálné látky metastabilní stav – skelný přechod

monoklinic

Za 1Atm
a nízkého
tlaku

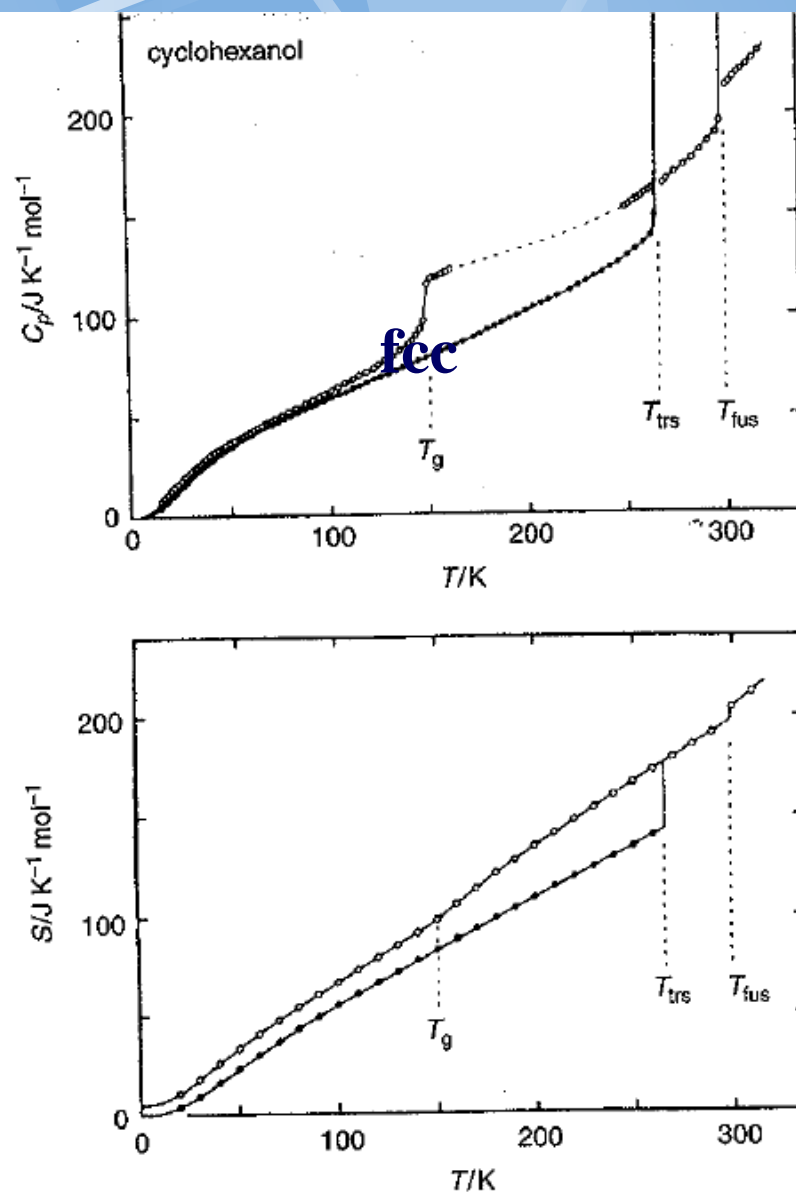


Figure 2 Heat capacity and entropy of cyclohexanol

Odhad K_0

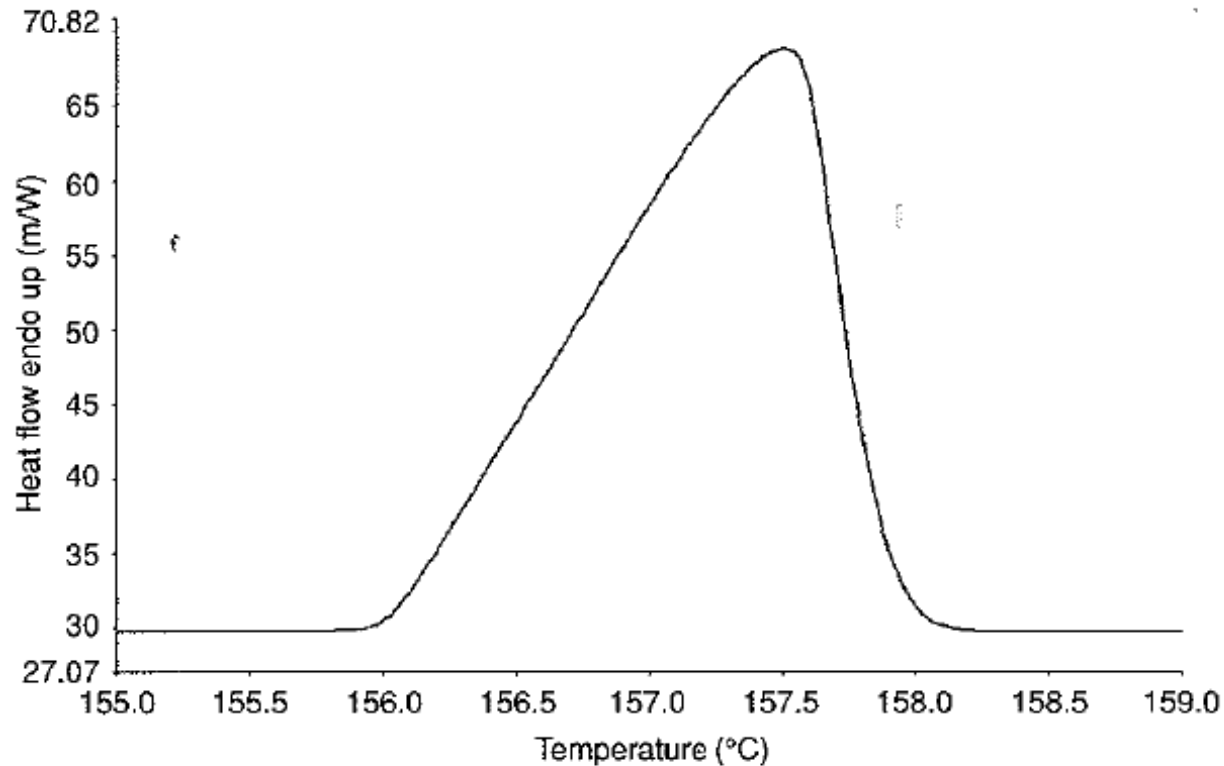
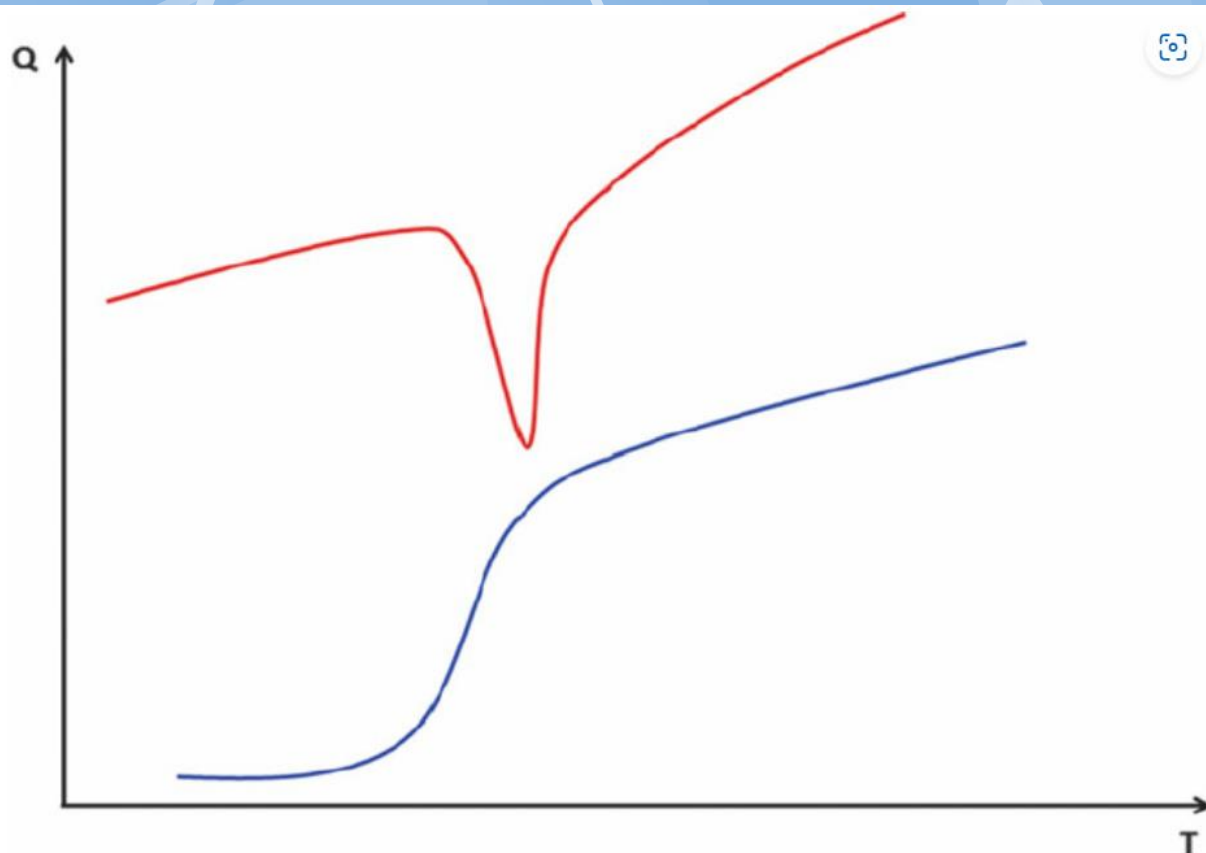


Figure 1.5 Indium heated at 5°C/min showing an almost triangular melting profile typical of single crystal melt at lower scan rates. The slope of the leading edge of the melt of a pure material such as indium gives a value for the thermal resistance constant R_0 .

DSC – phase transformation 1st and 2nd order



The typical DSC diagram for solid state phase transition with latent heat (red plot) or without latent heat (blue plot). The scale is not the same; in general the curve for a second-order transition (blue plot) is associated with smaller changes of heat capacity (and therefore more difficult to detect)

[\(PDF\) Cryo-Crystallography: Diffraction at Low Temperature and More \(researchgate.net\)](#)

Phase transformation studies (Examples)

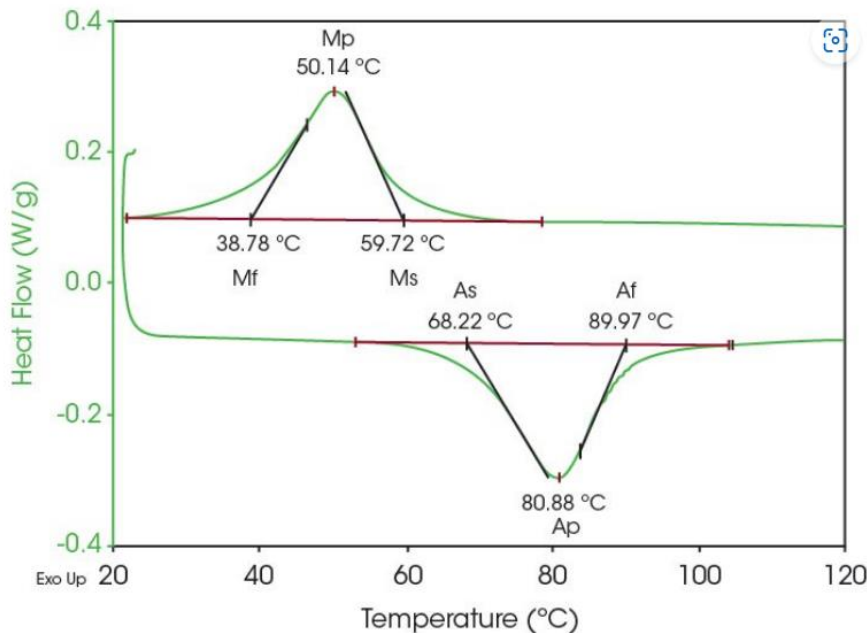


Figure 1. Austenitic and Martensitic thermal transitions in nitinol.

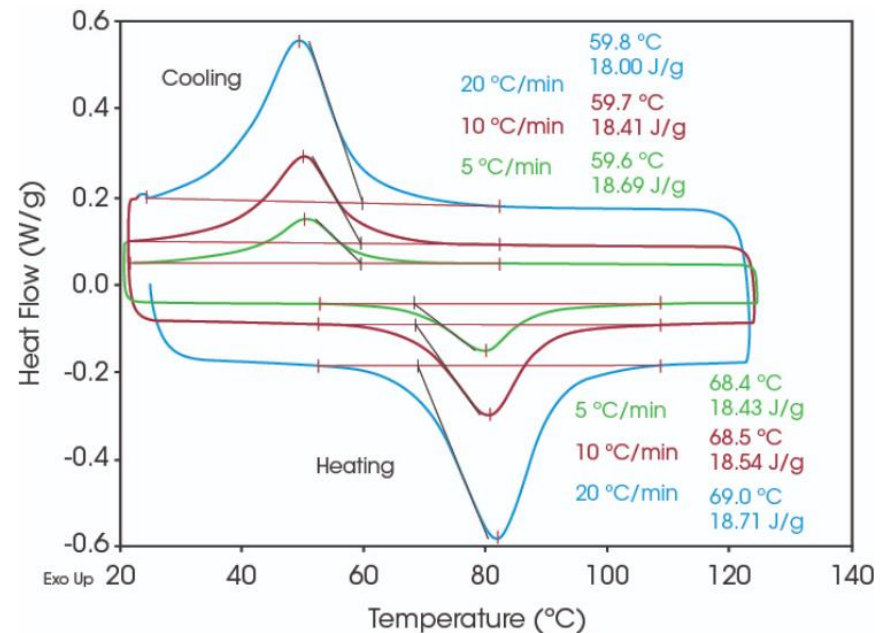


Figure 2. Effect of heating/cooling rate on nitinol transition.

A Study of the Nitinol Solid-Solid Transition by DSC - TA Instruments

