Audio test:

Termická analýza

Speciální metody TA

Přednášející: Doc. Jiří Sopoušek

Obsah

- Triple cell DSC (3cDSC)
- Knutsen cell
- Termomechanická analýza

Triple cel DSC

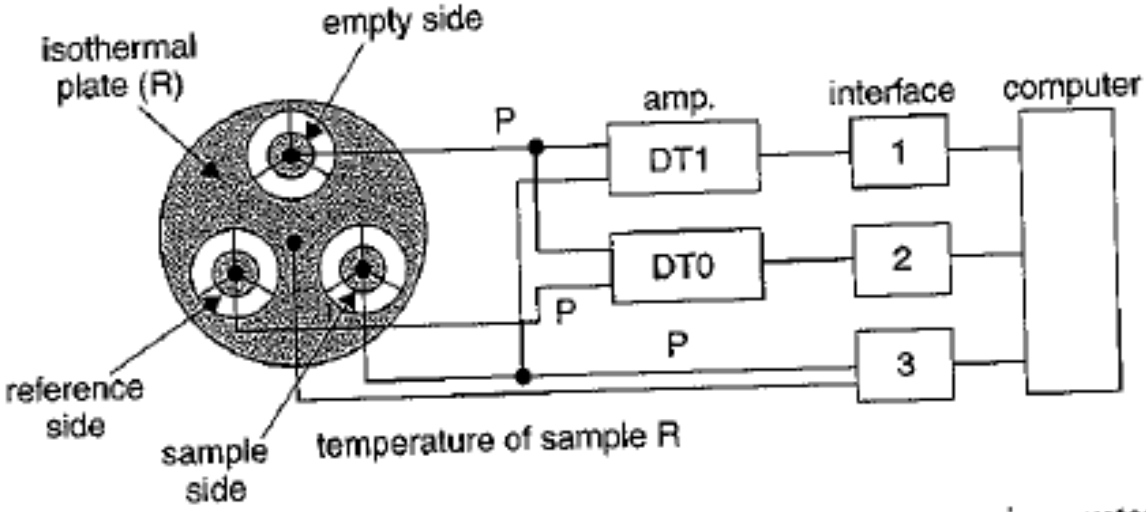


Figure 1 Block diagram of the cell arrangement and temperature-sensing system of the triple-cell DSC

$$C_s = C_r \cdot \frac{R_r}{R_s} \cdot \frac{\Delta_s T - (\Delta_s T)_{blank}}{\Delta_r T - (\Delta_r T)_{blank}}$$

(R_r/R_s) gives the calibration constant, k.

Stabilita - /+0,1K

Blank…Referenční měření s prazdnými pozicemi S a R

Měří se dvakrát: Blank a s držákem obsazeným sample a referencí.

Postup měření

Kalibrace na Al2O3 (0-1200stC), Měření blanku, měření s S+R.

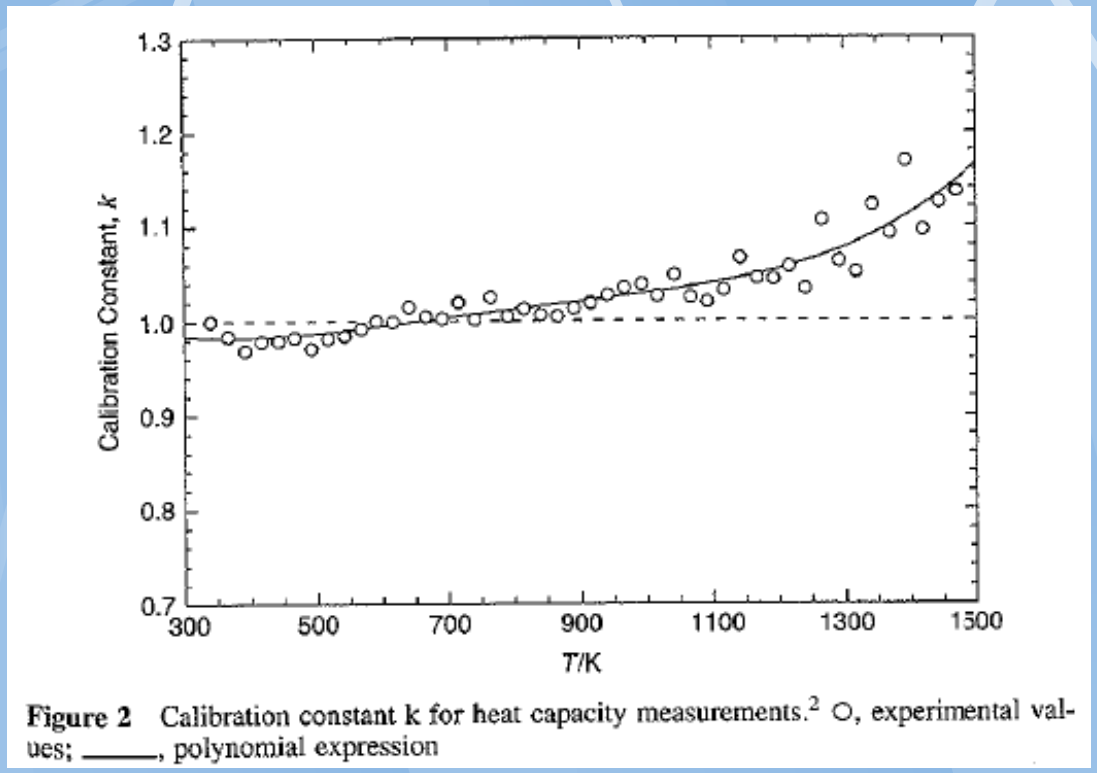


Figure 2 Calibration constant k for heat capacity measurements.[2] ○, experimental values; _____, polynomial expression

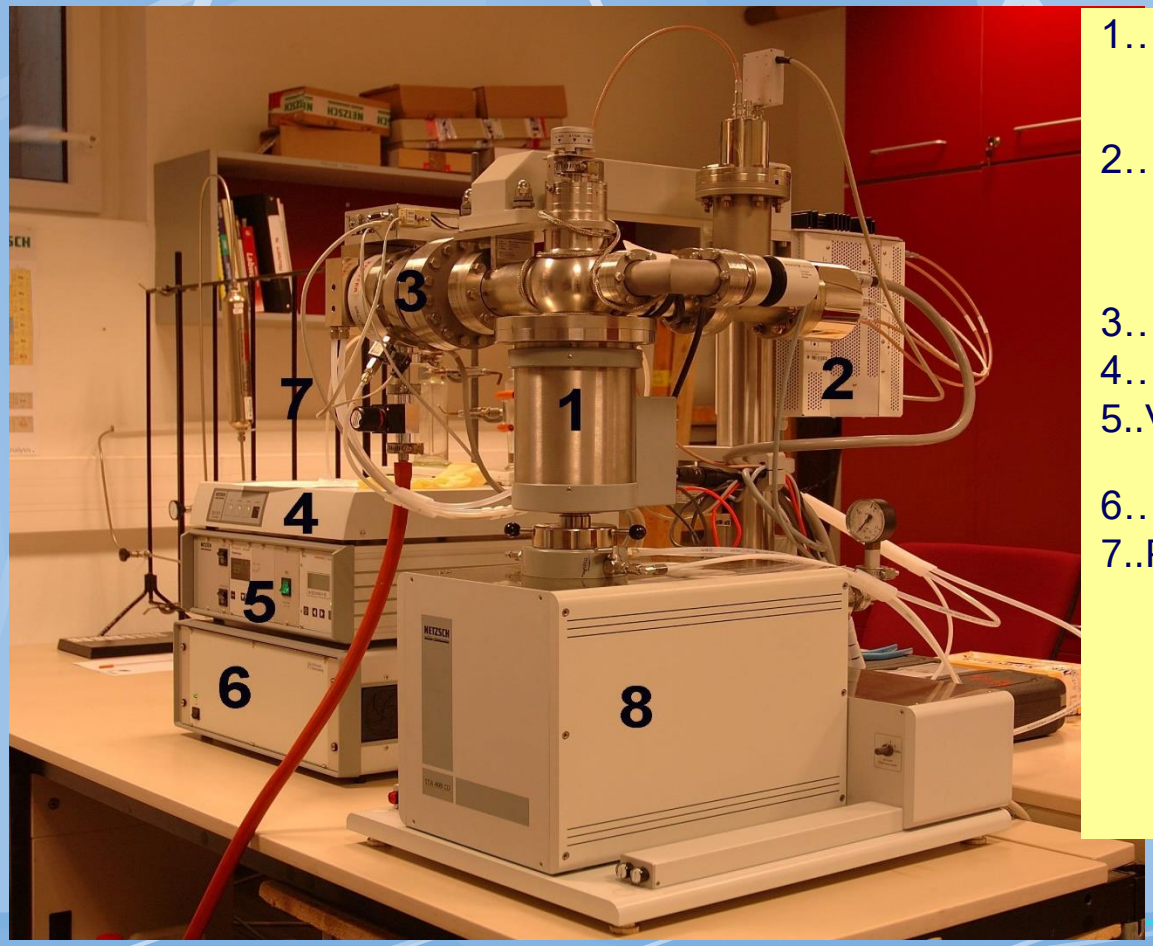
Měření Cp kovů:

±3 %

Měření kinetiky

http://www.tainstruments.com/main.aspx?siteid=6&id=289&n=1 (multi cell)

Knudsen cell (KEMS)



1…Furnace
(0.1 – 20 K min^{-1},
25-1450ºC)

2…QMS
range 1-512 amu
resolution 0,5amu
IE = 25 -100 eV

3…Turbomolecular Pump

4…**TA S**ystem **C**ontroller (TASC)

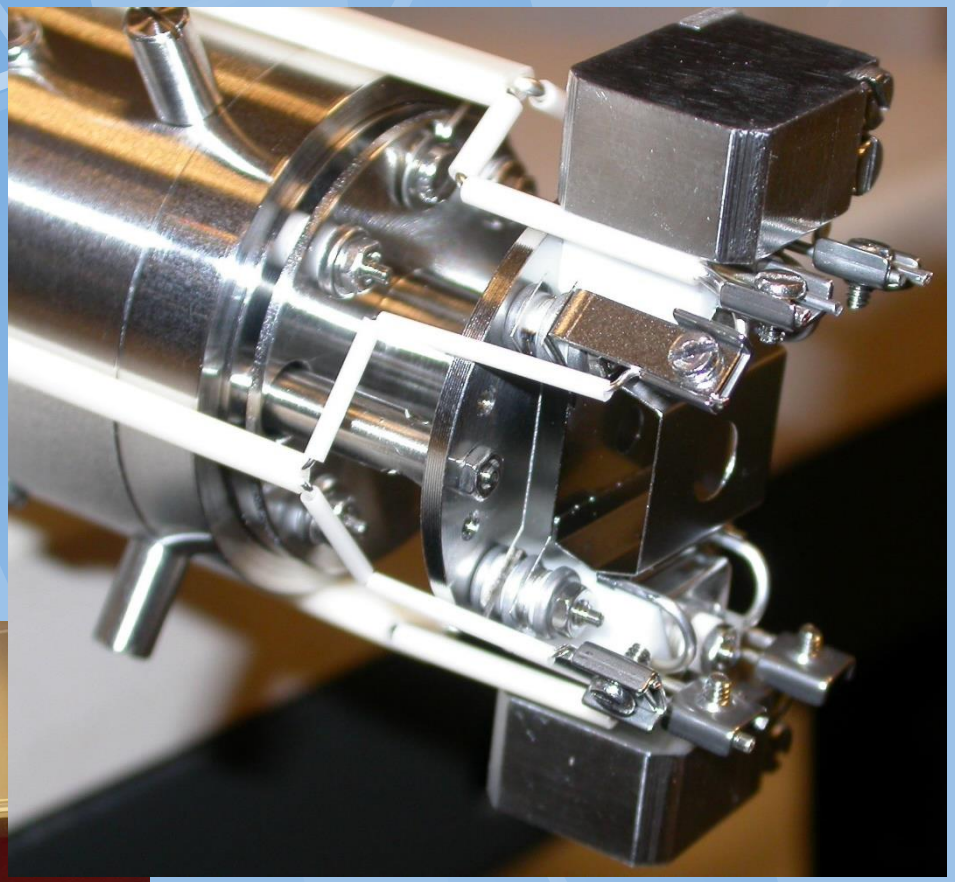
5..Vacuum Controller, (cca $9 \cdot 10^{-6}$ mbar)

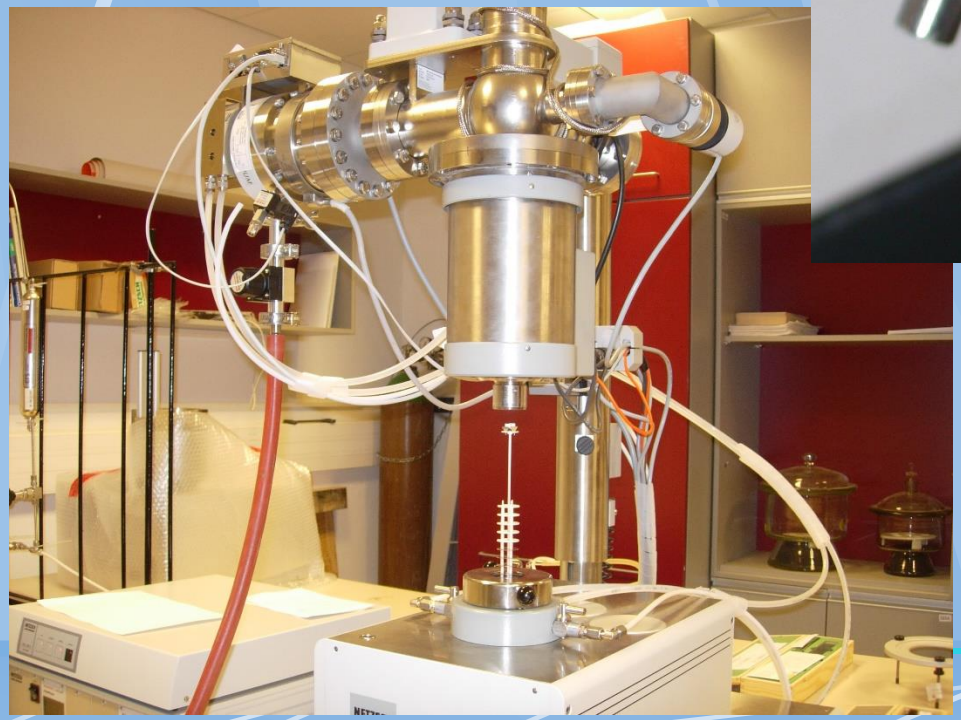
6…QMS Controller

7..Purification Column (oxygen)
(Argon 99,999)
Mass **F**low **C**ontroller (MFC)

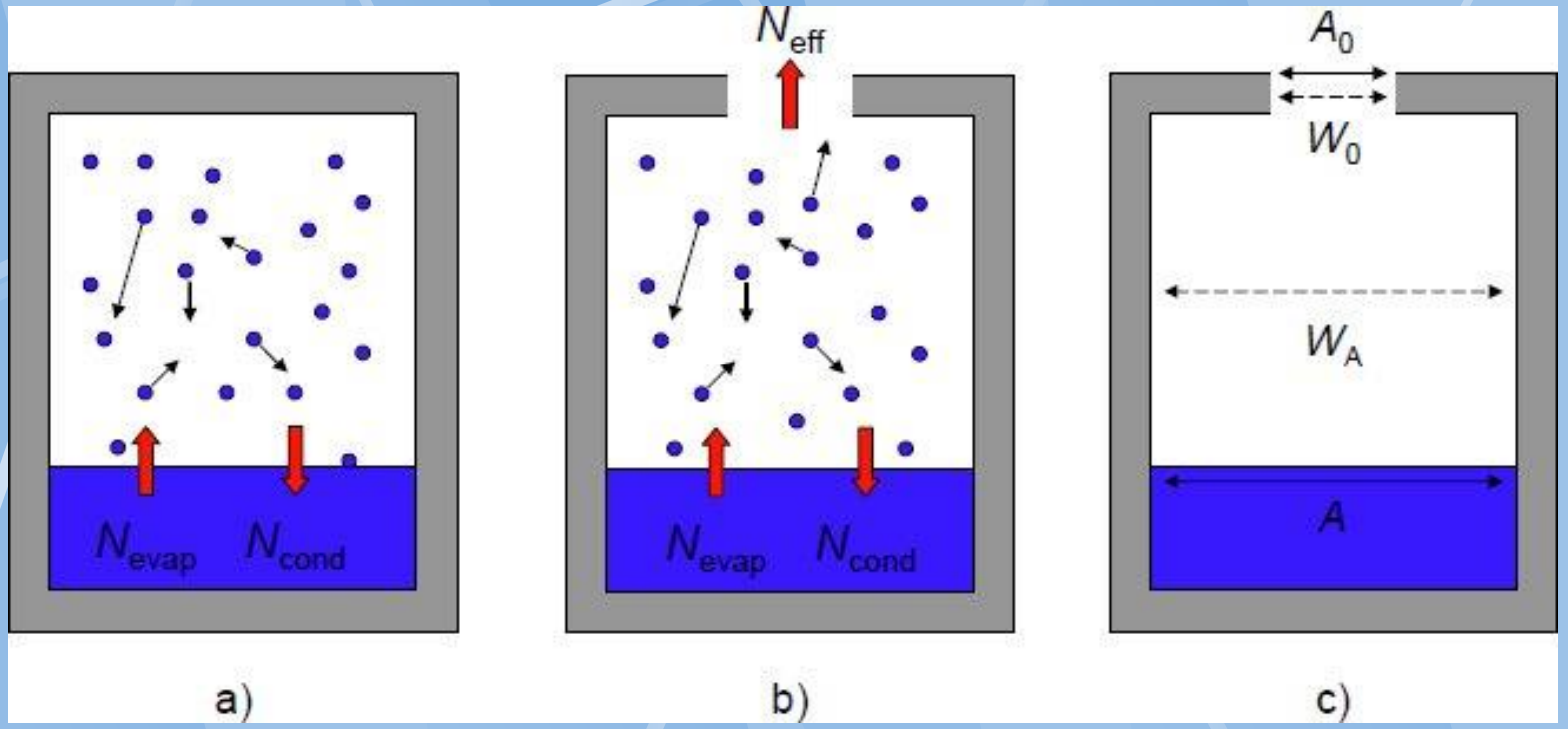
Knudsen cell / mass spectroscopy

STA 409 CD/3/403/5/G - detaily





Princip



Kinetická teorie plynů:

$$p_{\mathrm{meas}} = \frac{1}{W_0 A_0} \frac{dm}{dt} \sqrt{\frac{2\pi k_{\mathrm{B}} T}{M}}$$

dm/dt… hm. Ztráta (mikrováhy)

Whitman-Motzfeld equation

$$p_{\mathrm{eq}} = \left[1 + \frac{W_0 A_0}{A}\left(\frac{1}{\alpha} + \frac{1}{W_{\mathrm{A}}} - 2\right)\right] p_{\mathrm{meas}}$$

W_0…Clausiusův pravděpod. faktor (0-1) projití otvorem, α…pravděp. Kondenzace (pro kovy=1)

http://www.kip.uni-heidelberg.de/AG_Pucci/Research/LaboratoryAstrophysics/Experiments/Evaporation/Evaporation.php

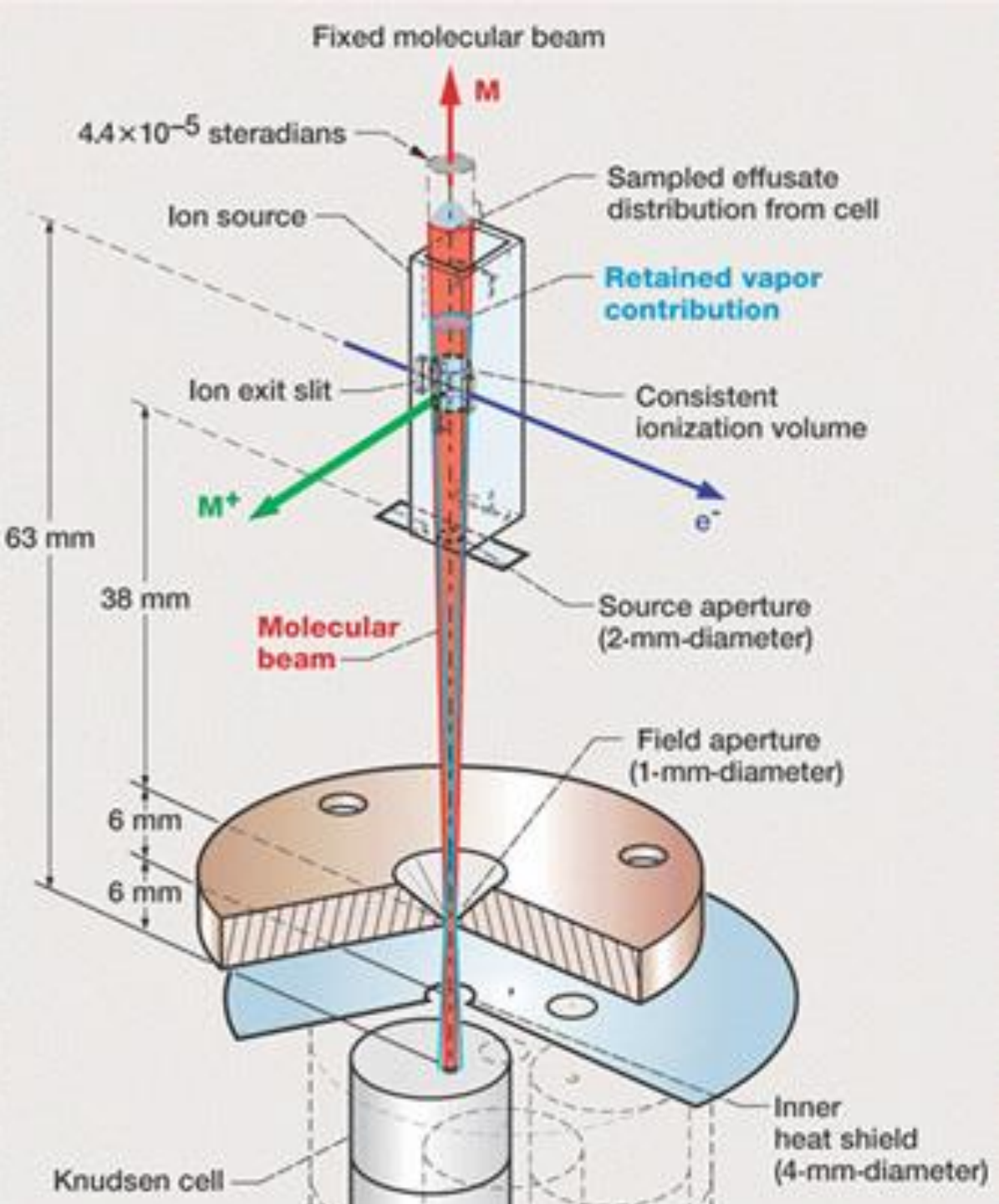
MS detekce více složek

Parciální tlak:

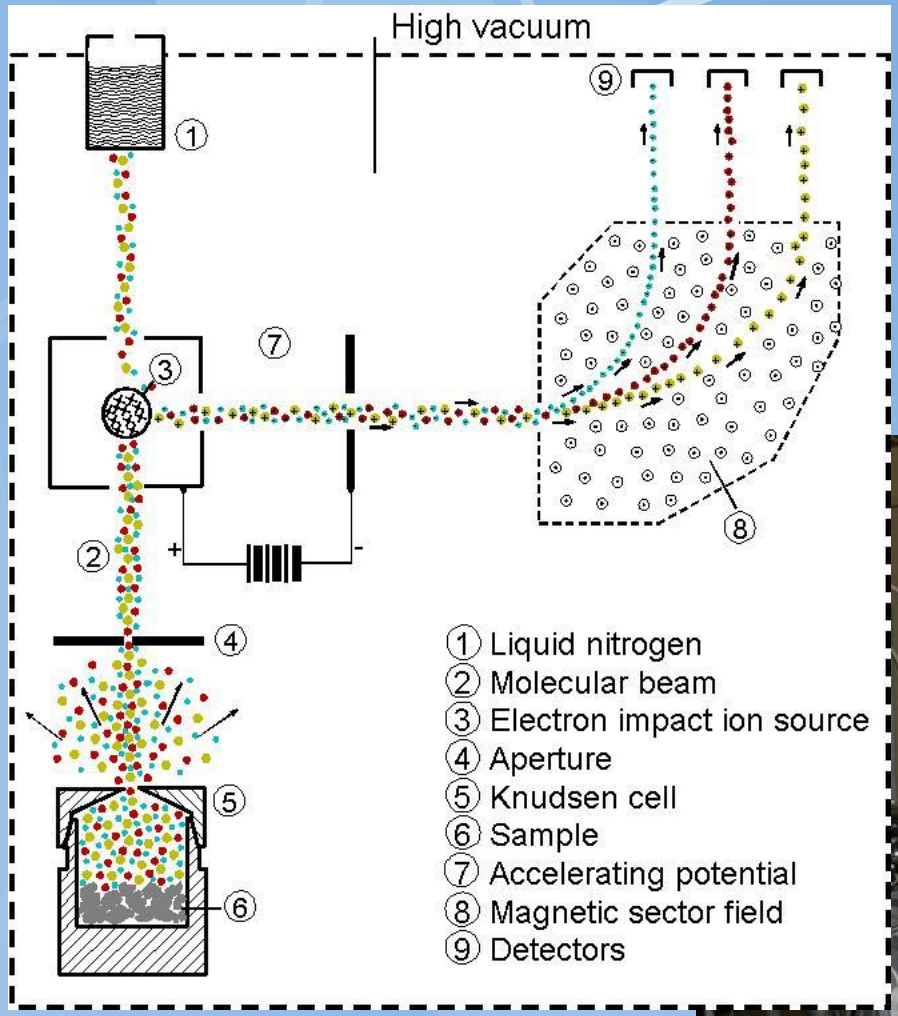
$$p_j = (I_{jk}^{+}\, T) / S_{jk}$$

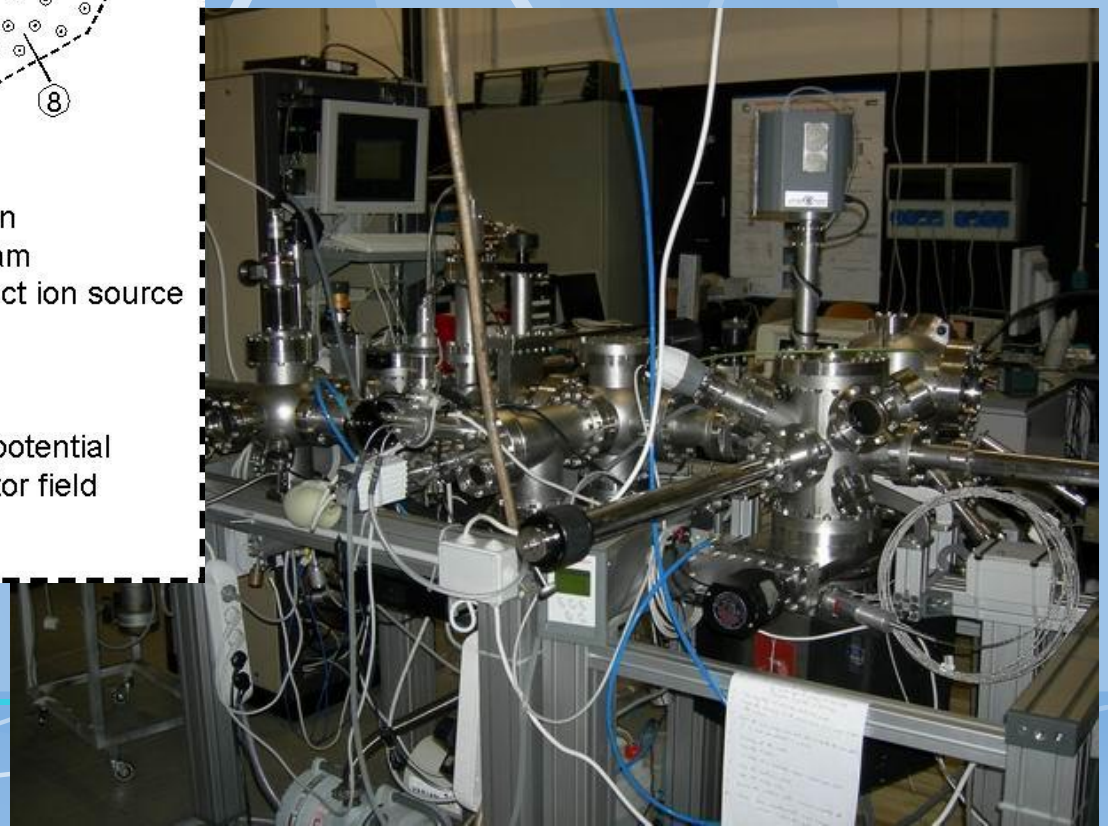
Aktivita složky:

$$a_j = P_j(A) / P_j(R) = [I_{jk}^{+}(A)\cdot T)] / [I_{jk}^{+}(R)\cdot T)] \cdot [S_{jk}(R)] / [S_{jk}(A)] = [I_{jk}^{+}(A)] / [I_{jk}^{+}(R)]$$



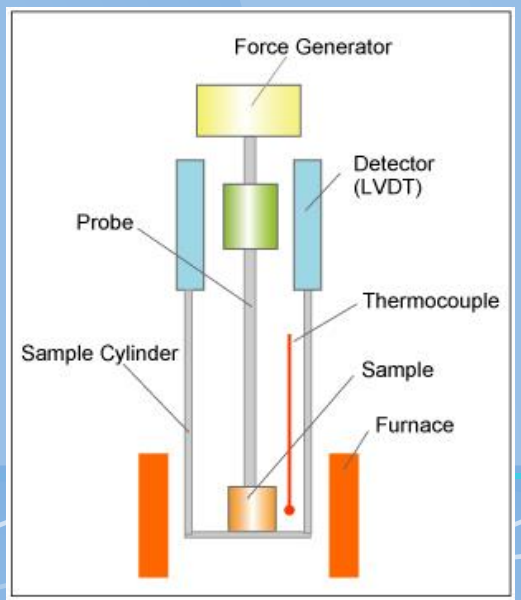
Praktické provedení

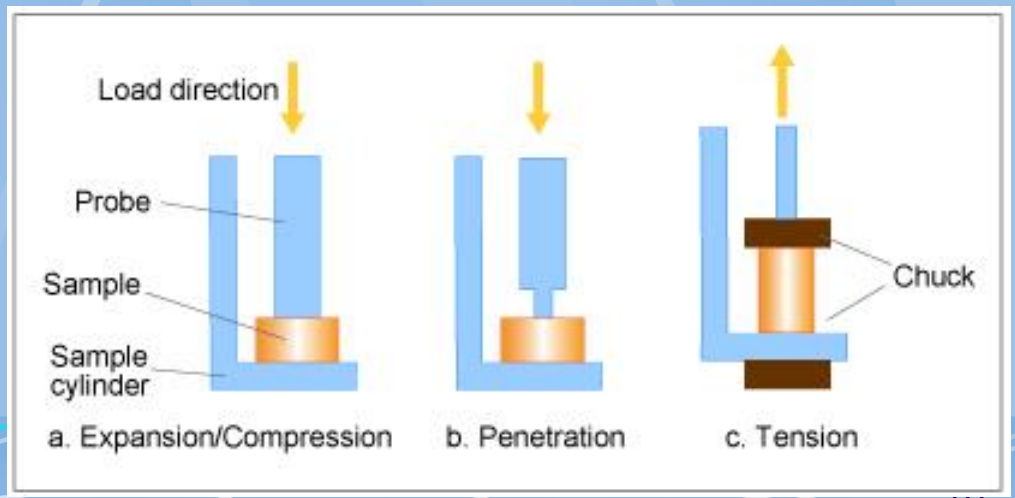




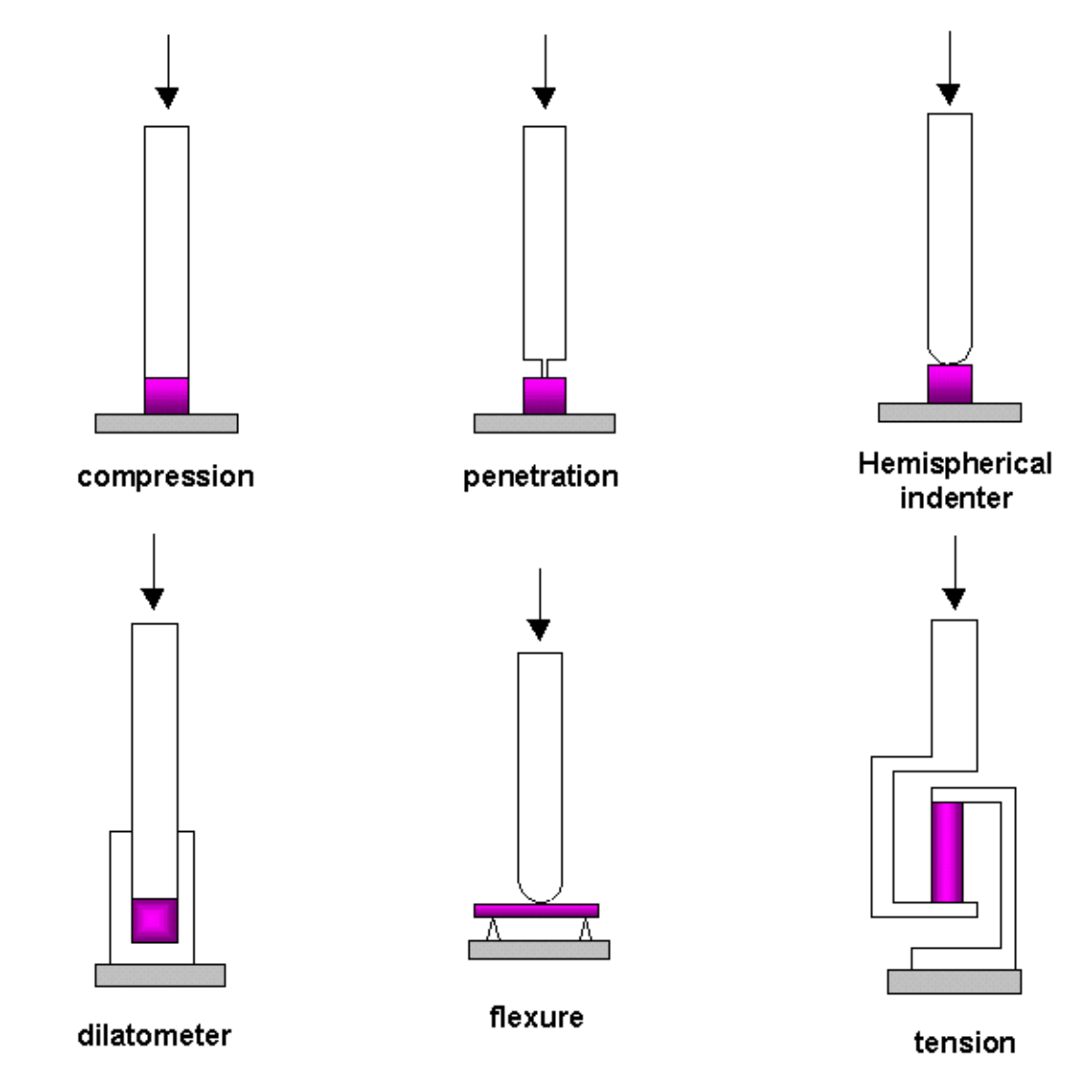
Termomechanická analýza (TMA)

ICTAC: **A technique in which a deformation of the sample under non-oscillating stress is monitored against time or temperature while the temperature of the sample, in a specified atmosphere, is programmed. The stress may be compression, tension, flexure or torsion.**





Variace mechanického namáhání



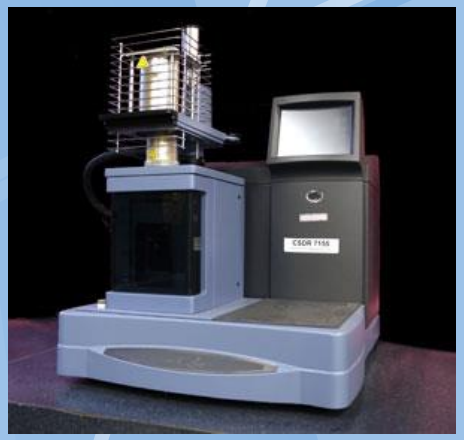
Hlavní použití:

Polymery

použití

Method	Mode	Measured Quantity	As a function of	Information Obtained
Bulk sample	Flat probe/light load	expansion	Temperature	Coefficient of Expansion and Tg
Divided sample	Dilatometer	Volumetric changes	Temperature	Coefficient of expansion and Tg
Thin film	Penetration probe/ Significant load	Depth of penetration	Force	Modulus, Cross-link density
			Time	Creep behaviour Cure behaviour
			Temperature	Softening (Tg) Melting
Film or fibre	Tension accessory	Uniaxial extension or shrinkage	Force	Modulus, Cross-link density
			Time	Creep behaviour, Cure behaviour
			Temperature	Tg, melting, Cure behaviour, Prepn. History
Fluid	Parallel plates	Distance	Time	Viscosity, Gelation
			Temperature	Melting, Viscosity, Gelation
Bulk or supported	Flexure accessory	Bending	Time	Creep behaviour
			Temperature	Softening (Tg) Melting

Přístroje

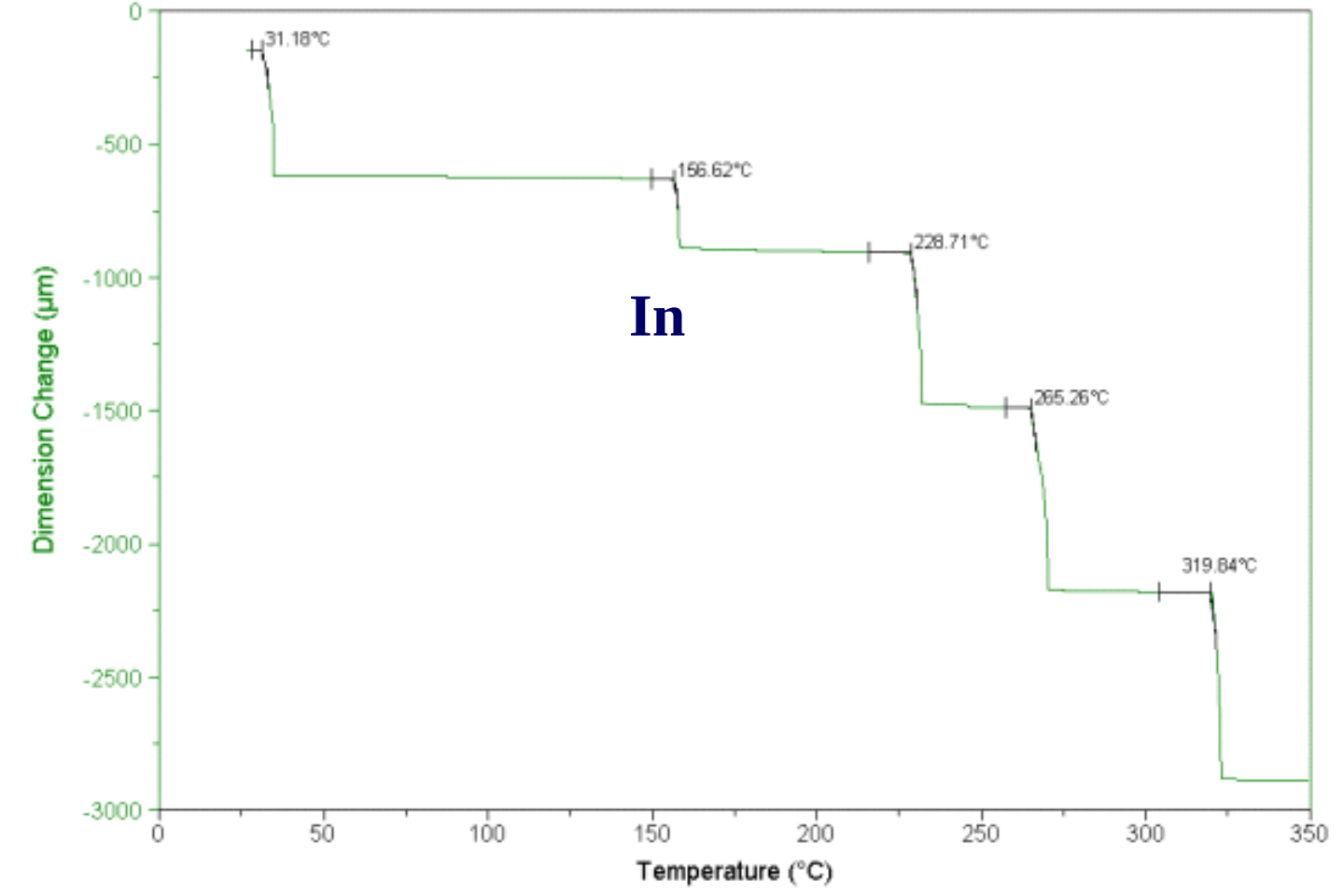


ASTM E831

Linseis

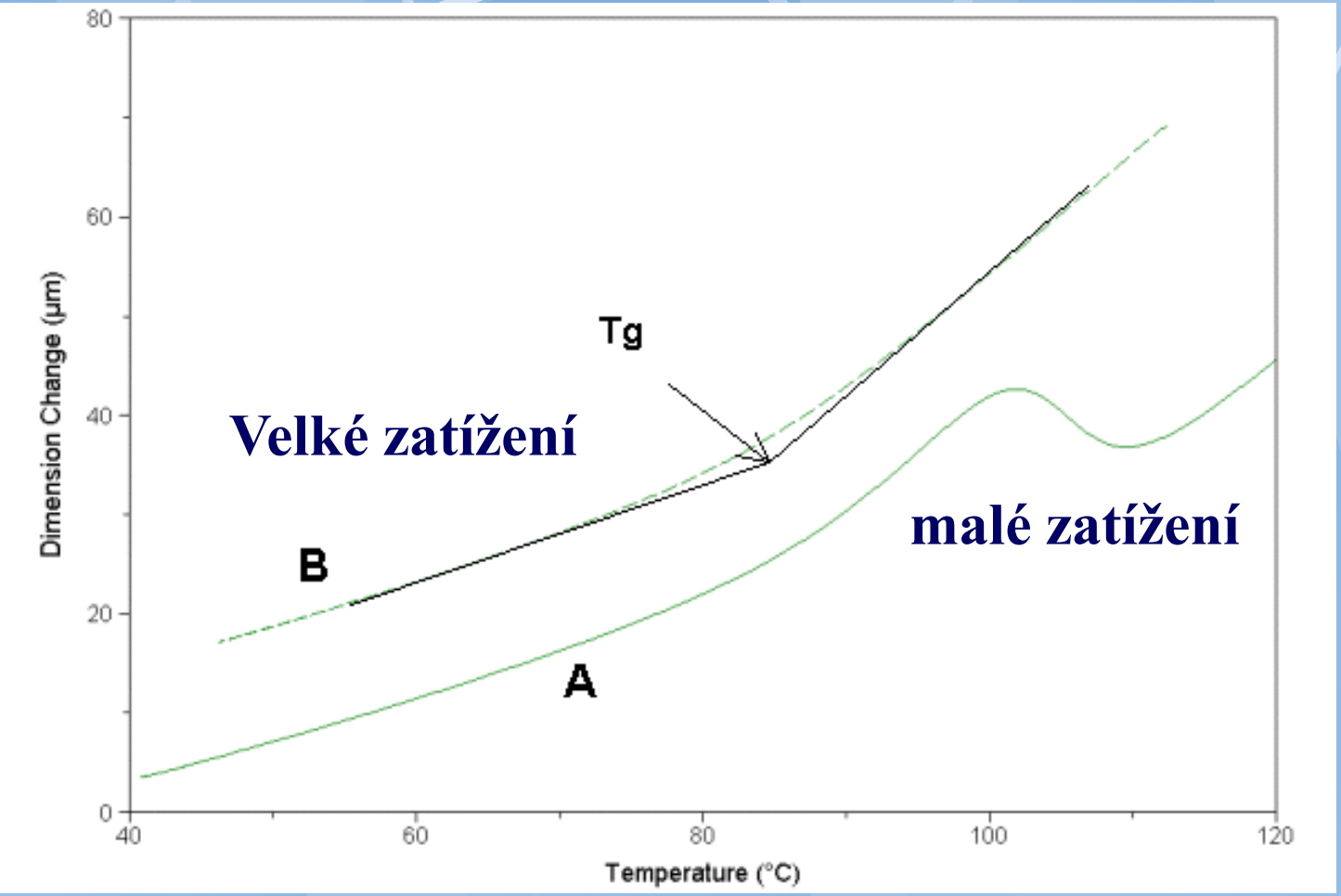
Dilatometer Netzsch 402

Kalibrace na teplotu



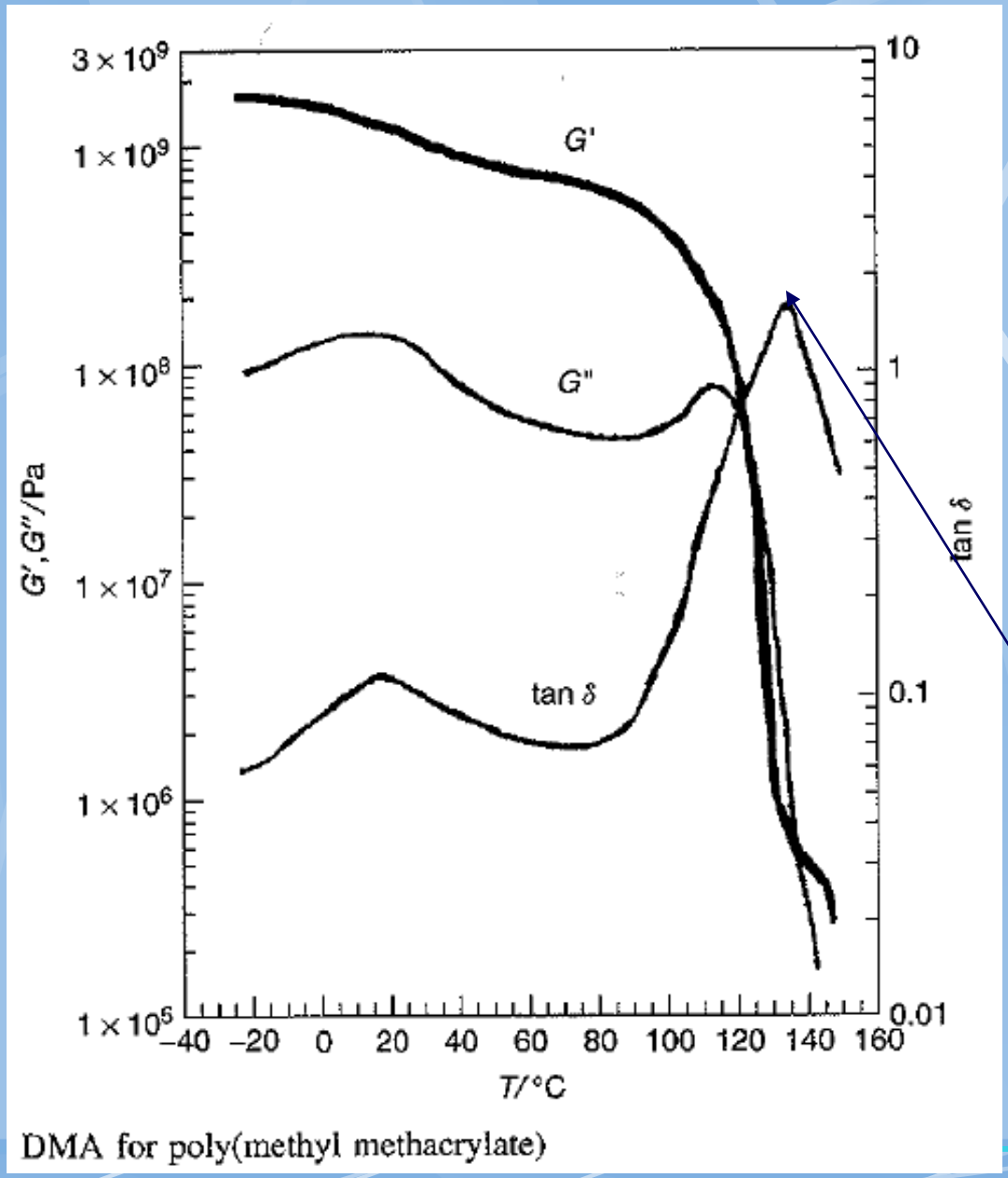
Mnohonásobný sendwič z čistých kovů

Aplikace



Určení teploty skelného přechodu Tg PTFE

Reaktivita



DMA for poly(methyl methacrylate)

G^{I} Storage modulus

G^{II} Loss modulus

Uvolnění - _COOCH3 ramen

TBA torsional braid analysis

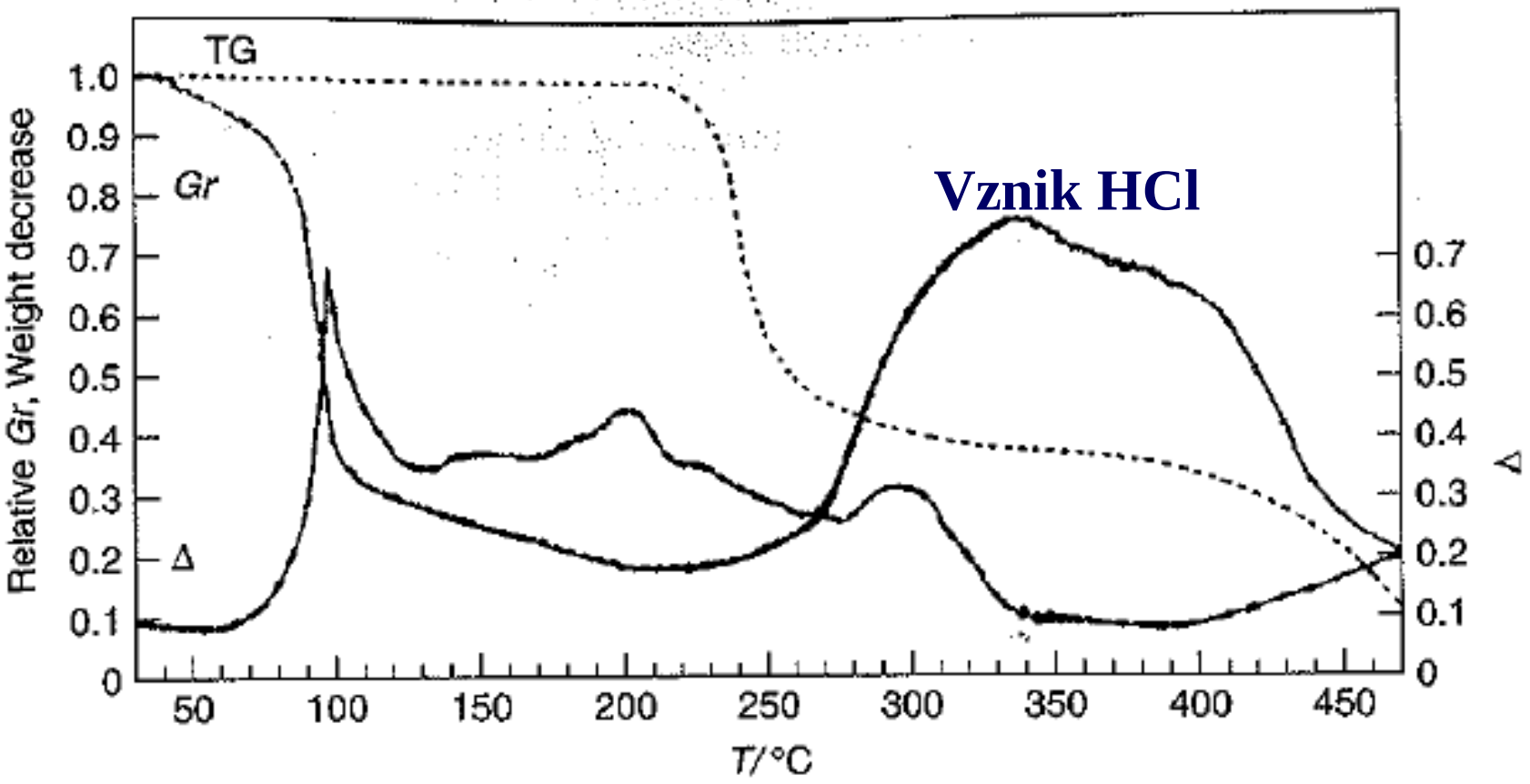


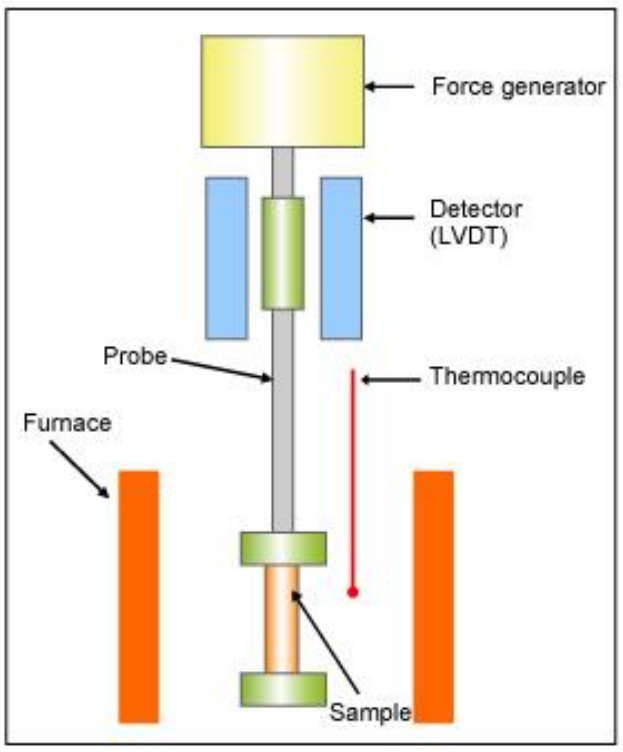
Figure 4 TBA for poly(vinyl chloride)

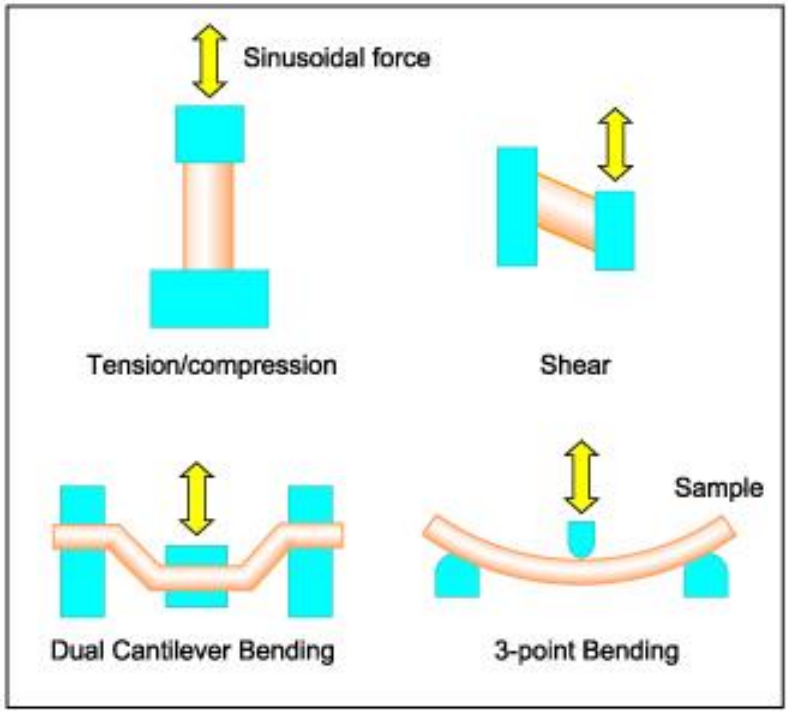
Dynamická mechanická analýza

- A technique in which the sample's kinetic properties are analyzed by measuring the strain or stress that is generated as a result of strain or stress, varies (oscillate) with time, applied to the sample.

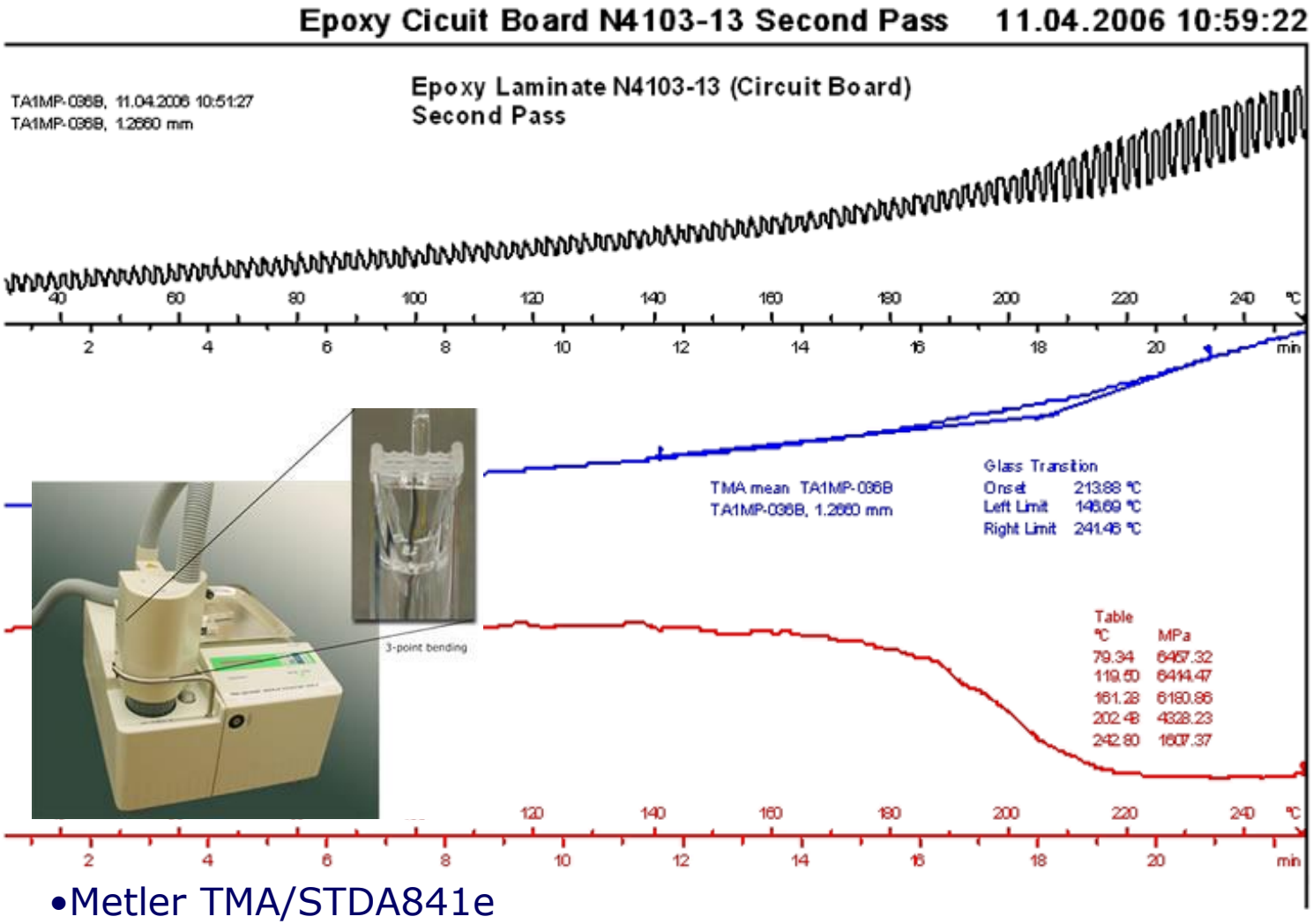
- Static Viscoelasticity Measurement

A technique in which the change in stress or strain is measured under uniform stress or strain that remains constant across time.





Cyklické zatížení (Tg)



- Metler TMA/STDA841e

Diskuse

Reálné látky metastabilní stav – skelný přechod

monoklinic

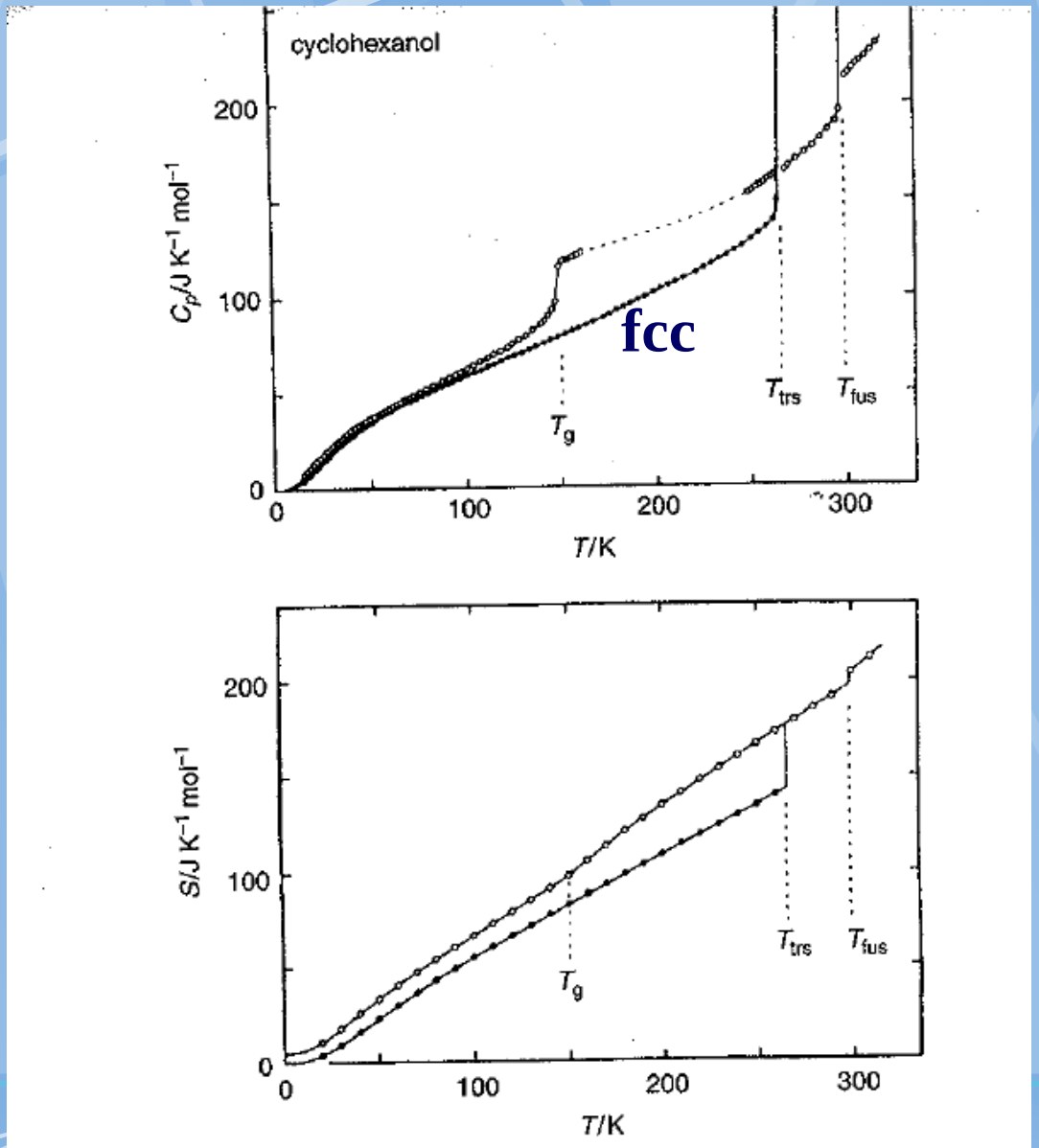
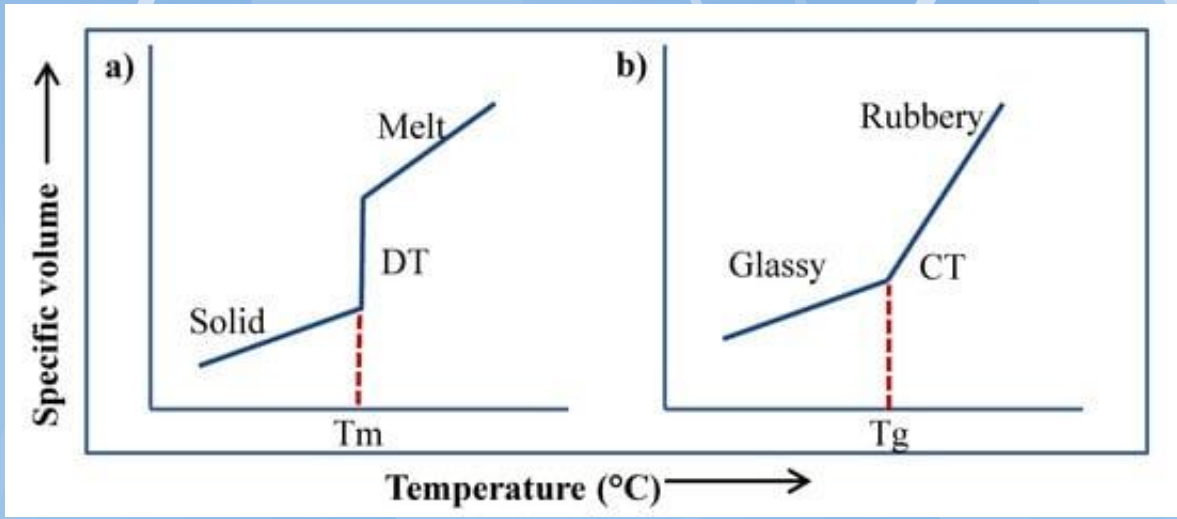


Figure 2 Heat capacity and entropy of cyclohexanol

Za 1Atm a nízkého tlaku

Dilatometry

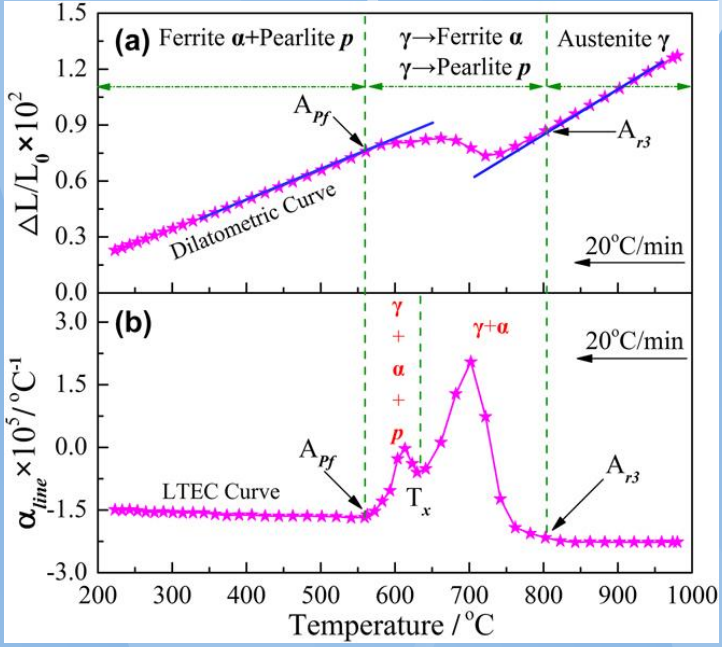
Volume changes of phase transformations



Schematic representation of first and second-order transitions. (a) Melting transition of a crystalline solid. (b) Glass transition of an amorphous solid.

Polymers | Free Full-Text | Application of Differential Scanning Calorimetry (DSC) and Modulated Differential Scanning Calorimetry (MDSC) in Food and Drug Industries (mdpi.com)

Experimental evaluation of transformation - dilatometry



Modulated Differential Scanning Calorimetry (MDSC)