Differential Thermal Analysis (DTA) Differential Scanning Calorimetry (DSC)

Characterisation methods of inorganic materials

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Goal of the lecture

To learn the main operating principle of the Differential Thermal Analysis (DTA) and Differential Scanning Calorimetry (DSC)

- What is measured? How?

To learn how DTA and DSC can be used in (metallurgical) R&D

- How the results can be used?
- How the results of DTA/DSC and TGA support each other?

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Contents

Differential Thermal Analysis - DTA

Differential Scanning Calorimetry - DSC

- Main principles
  - What is measured?
  - What kind of phenomena can be observed?
- Strengths and restrictions
  - Requirements for samples
- Connection with other devices

Utilization of DTA/DSC results

- Application areas in (metallurgical) R&D
- How the results can be processed further?

Examples

Differential Thermal Analysis (DTA)

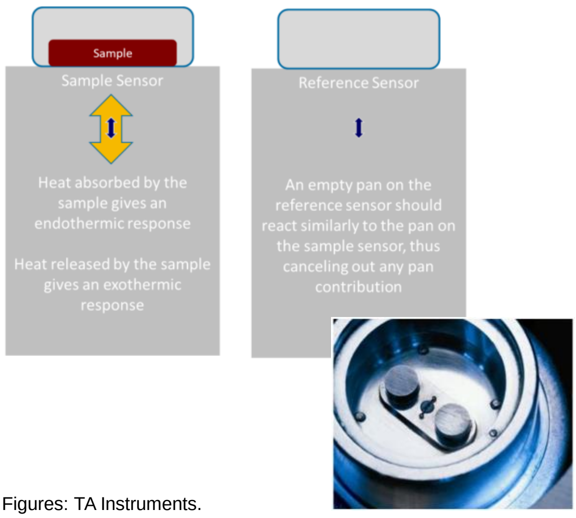
Main operating principle

- Temperature difference between sample and a reference is measured as a function of time
  - Heating and/or cooling at a certain rate
    - Results often shown as a function of temperature rather than time
  - (Isothermic tests)
  - Atmosphere is chosen to correspond the conditions of interest
    - Reducing, oxidising, inert, sulphurizing, *etc.*
    - Simulation of certain process conditions
    - CO, CO_2, H_2, H_2O, Ar, N_2, O_2, S_2, SO_2, *etc.*

All the phenomena in which entalphy / heat content changes can be observed

- Chemical reactions (exo-/endothermic)
- Removal of moisture and/or volatile components (endoth.)
- Phase transformations (endoth. when heated)
  - No mass change - not observable with TGA

Differential Scanning Calorimetry (DSC)



Figures: TA Instruments.

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- Phase transformations (endoth. when heated)
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N.B. From this point onwards the presentation focuses on DSC, but most things are applicable for DTA, too.

Differential Scanning Calorimetry (DSC)

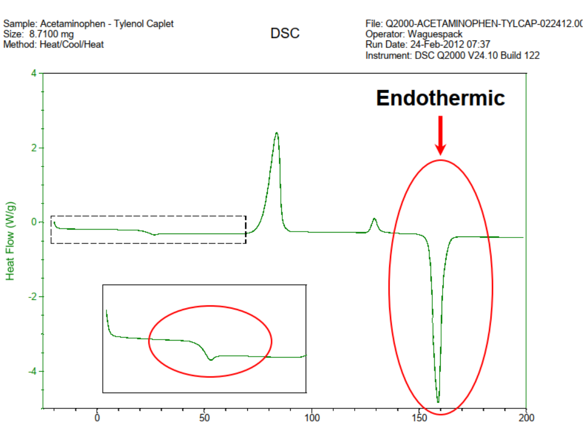

Figure: TA Instruments.

Observation of endothermic phenomena

- Heat flows into the sample as a result of

- heating	heat capacity	C_P
- melting	enthalpy of fusion (melting)	ΔH_F / ΔH_M
- evaporation	enthalpy of vaporization (boiling)	ΔH_V / ΔH_B
- sublimation	enthalpy of sublimation	ΔH_S
- phase transformation (during heating)	enthalpy of transformation	ΔH_{TR}
- glassy transition ("melting" of amorphous materials)		
- reaction	enthalpy of reaction	ΔH_R
- For endothermic reactions such as reduction		
- mixing	enthalpy of mixing	ΔH_M
- For endothermic mixing of components		

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Differential Scanning Calorimetry (DSC)

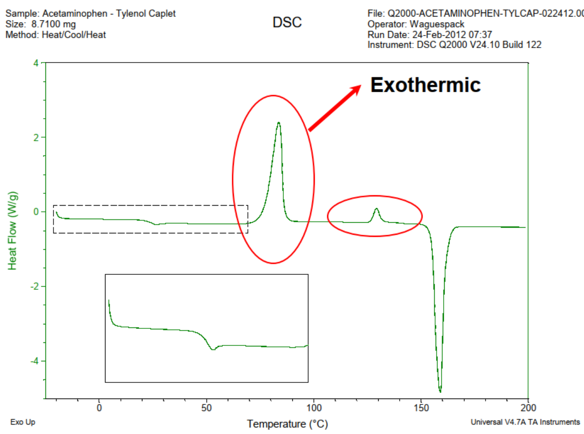


Figure: TA Instruments.

Observation of exothermic phenomena

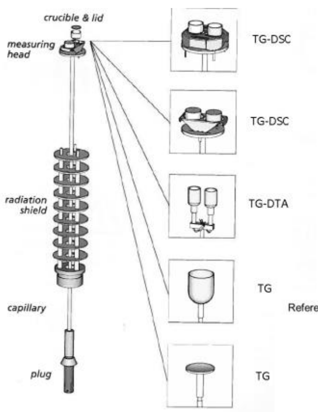
- Heat flows out of the sample as a result of
  - cooling — heat capacity — $-C_P$
  - solidification — enthalpy of fusion (melting) — $-\Delta H_F$ / $-\Delta H_M$
  - condensation — enthalpy of vaporization (boiling) — $-\Delta H_V$ / $-\Delta H_B$
  - deposition — enthalpy of sublimation — $-\Delta H_S$
  - phase transformation (during cooling) — enthalpy of transformation — $-\Delta H_{TR}$
  - crystallisation (of amorphous materials)
  - reaction — enthalpy of reaction — ΔH_R
    - For exothermic reactions such as oxidation / combustion
  - mixing — enthalpy of mixing — ΔH_M
    - For exothermic mixing of components

Differential Scanning Calorimetry (DSC)

Requirements for samples

- Size suitable for furnace/device
  - Fits crucible/sample basket (size often very small; 1...20 mg)
  - Challenge with representativity using very small sample size!
- Preferably in solid state
  - Molten samples may be studied in some cases
- Does not destroy the equipment
  - Does not react with the crucible/sample basket
  - Does not release gas components which are (too) corrosive for gas treatment equipment
    - e.g. alkali, chlorine and sulfur can be detrimental

Differential Scanning Calorimetry (DSC)



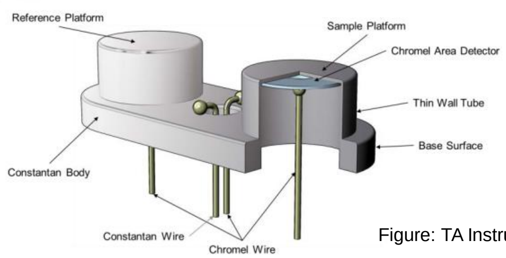


Figure: TA Instruments.

Sample baskets / Crucibles

- Different kind of crucibles made of different materials for different purposes (material, T, conditions, *etc.*)
  - Metals (e.g. Al, Cu, Au, Pt, stainless steel)
  - Ceramics (e.g. Al_2O_3)

Measurement of temperature

- Temperatures of sample and reference
  - With a thermocouple at the sample platform
  - No direct contact between thermocouple and sample
- Programmed temperature and furnace temperature are usually not recorded
  - Program temperature used for furnace control
  - Furnace temperature defines sample/reference temperatures

Differential Scanning Calorimetry (DSC)

Figure: Riku Mattila.

To obtain more detailed/informative data:

1) DSC (or DTA) is often connected with other devices

- Thermal Gravimetric Analysis
- Offgas from the system may be analysed (e.g. MS)

2) Samples are often characterized after tests

- Chemical composition
- Mineralogical composition
- Other properties (*e.g.* strength, leaching, *etc.*)

3) Results may be further processed

- Determination of thermochemical properties
  - Heat capacity
  - Entalphy
- Rate phenomena (time-dependence) may be studied
  - Determination of kinetic parameters from the results of isothermic experiments in at least three different temperatures

Differential Scanning Calorimetry (DSC)

Example of results and their interpretation

- Observation of melting and vaporization from TGA and DSC curves

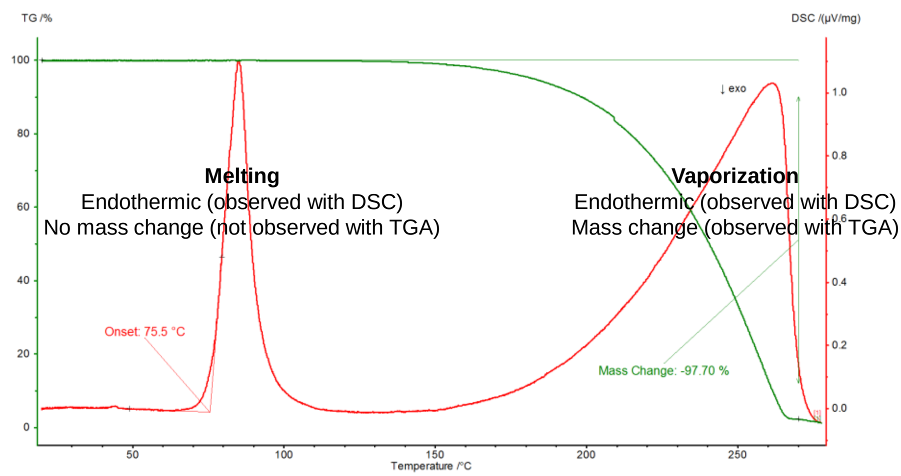


Figure from: Tommi Kokkonen.

Differential Scanning Calorimetry (DSC)

Example of results and their interpretation

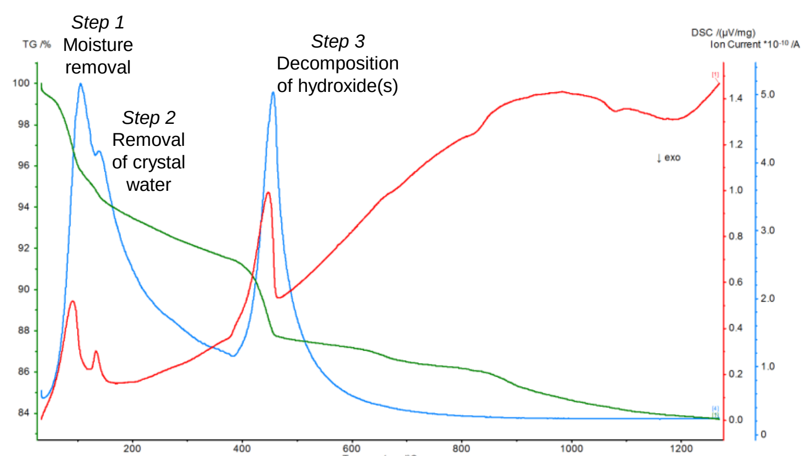
- Observation of H_2O removal from TGA, DSC and MS curves

It is seen from the TGA curve (green) that mass of the sample is decreased in several steps.

These steps indicate either:
- removal of volatile compounds
- chemical reactions with gaseous reaction products.

It is seen from the DSC curve (red) that the above mentioned phenomena is:
- endothermic
- occurs clearly in three stages

It is seen from the MS curve of H_2O (blue) that water vapour is released in the above mentioned phenomena in three stages



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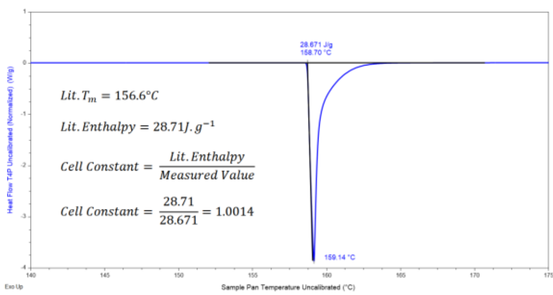
Differential Scanning Calorimetry (DSC)



Selection of Temperature & Enthalpy Standards

- Benzoic acid (147.3 J/g) T_m = 123°C
- Indium (28.71 J/g) T_m = 156.6°C

Enthalpy (cell constant)

- Cyclopentane = -93.43°C
- Cyclohexane = -83°C
- Adamantane = -65.54°C
- Water = 0°C
- Gallium = 29.76°C
- Indium = 156.60°C
- Tin = 231.95°C
- Lead = 327.46°C
- Zinc = 419.53°C

Temperature

Figure and list of calibration standards: TA Instruments.

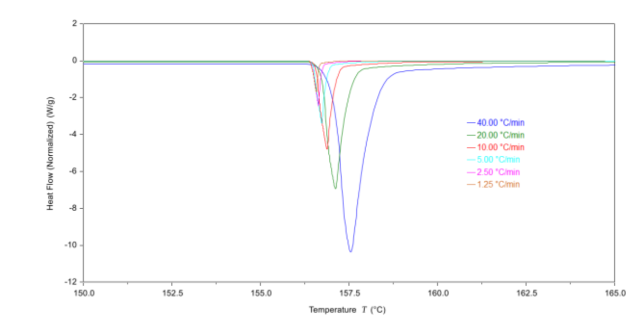
Restrictions

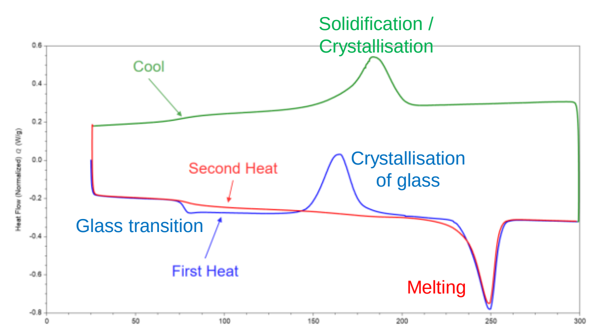
- Has to be calibrated with known samples
  - Temperature calibration typically with melting points of pure metals (e.g. indium, lead, *etc.*) as references
  - Heat flow calibration with standards of either heat of fusion/melting or heat capacity - Definition of cell constant
- Necessary to exclude the baseline from the results
  - Baseline run with an empty cell over the temperature range of interest
- Heating/cooling rate as well as possible atmospheres are limited
  - Depends on the equipment
- Sources of inaccuracy
  - Heating/cooling rate
    - System is not in spatial or temporal equilibrium
  - Sample size limitations
    - Small samples not representative
    - Large samples not uniform in composition/conditions
      - Transport phenomena as limiting factors
  - Conditions in sample and reference are not completely identical, although they are assumed to be (temperature, thermal resistance, heat capacity, *etc.*)
  - Heat exchange with the surroundings should be zero
  - Can be decreased (but not completely removed) with better equipment (e.g. to obtain a thermal resistance imbalance of less than 1%, the manufacturing tolecance of sensors is 1.27μm)

Differential Scanning Calorimetry (DSC)

The results are influenced by *e.g.*:

- ”direction” of temperature change (heating/cooling)
- heating/cooling rate
- thermal history of the sample





Figures: TA Instruments.

Differential Scanning Calorimetry (DSC)

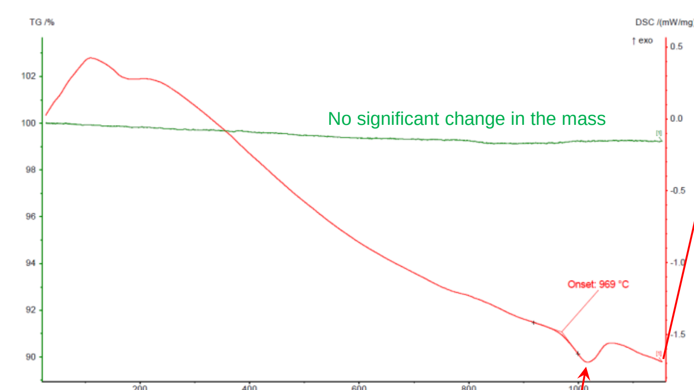
Possible areas of utilization

- Phase transformations
  - incl. solid state transformations and melting in which the mass of the sample does not change
  - Dynamic method for determining phase diagrams
- Removal of moisture/volatiles from materials
- Chemical reactions - *e.g.*:
  - Decomposition of compounds
  - Reduction of materials
  - Oxidation of materials
- Definition of thermochemical properties of materials
  - Entalphy, heat capacity
- Thermal stabilities of materials
- Characterisation of multicomponent systems
  - Comparison of experimental results with known behaviour of different elements/compounds

Example

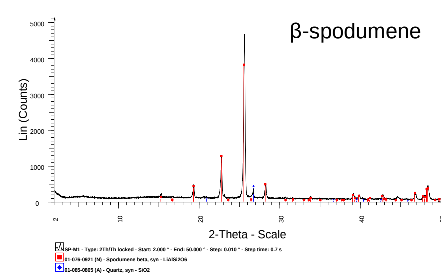
Observation of phase transformations

- Phase transformation of spodumene ($LiAlSi_2O_6$): $\alpha \rightarrow \beta$
  - Required as a pretreament before leaching of spodumene in the production of lithium-chemicals

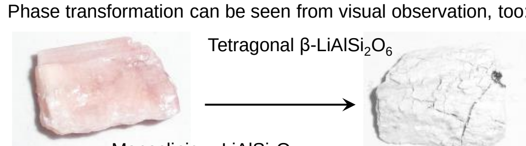


Figures from: Pekka Tanskanen.

Verification of the phase transformation with XRD:



Phase transformation can be seen from visual observation, too:



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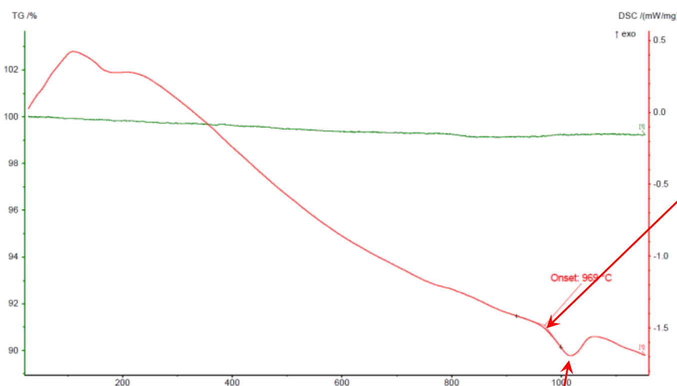

Temperature indicating the phase transformation temperature

The results of the DTA/DSC-analysis depend on the heating rate.
Slower rate → More uniform conditions
Faster rate → Transport phenomena have a bigger role

To eliminate the effect of the heating rate, the experiments should be executed with different heating rates and the results should be extrapolated to correspond the ”zero heating rate”.

Endothermic change from α-form into β-form

Figures from: Pekka Tanskanen.

Example

Observation of phase transformations

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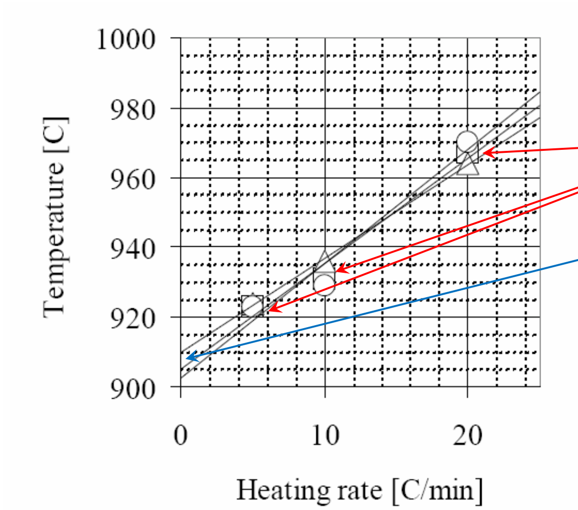

Observed phase transformation temperatures with different heating rates (5, 10 and 20 °C/min)

”Actual” phase transformation temperature which would be obtained with ”zero heating rate”

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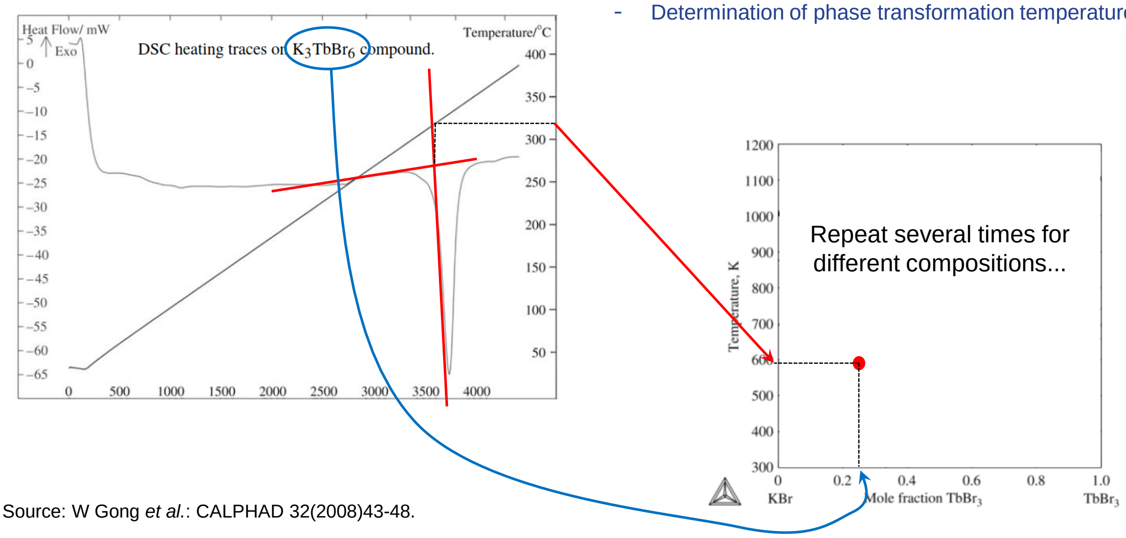
Figure: Tanskanen, Heikkinen, Karjalainen, Seppelin & Lassi: Proceedings of Eco-mates 2011. 28-30.11.2011. Osaka, Japan. pp. 219-220.

Example

Determination of phase transformations for phase diagrams

- Formation of K_3TbBr_6 in potassium-terbium-bromine system
- Determination of phase transformation temperature



Source: W Gong *et al.*: CALPHAD 32(2008)43-48.

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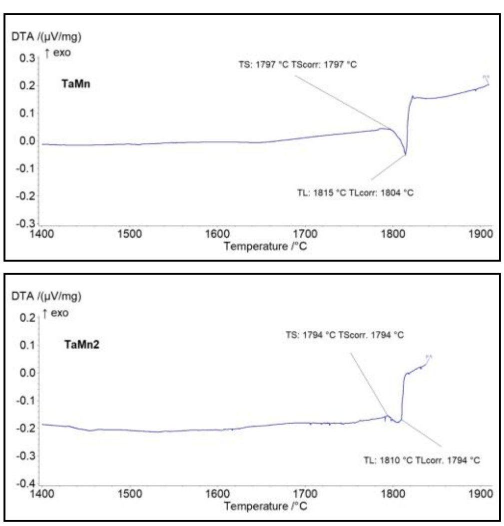
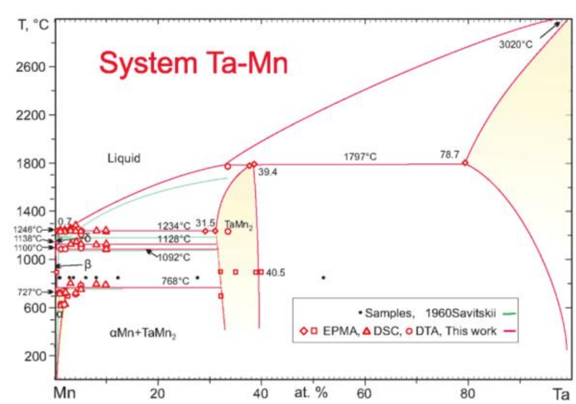
Example

Determination of phase diagrams

- Dynamic determination of Ta-Mn phase diagram using DTA/DSC (+ other thermochemical data)





Source: X Yan *et al.*: Journal of Alloys and Compounds. 865(2021)158715.

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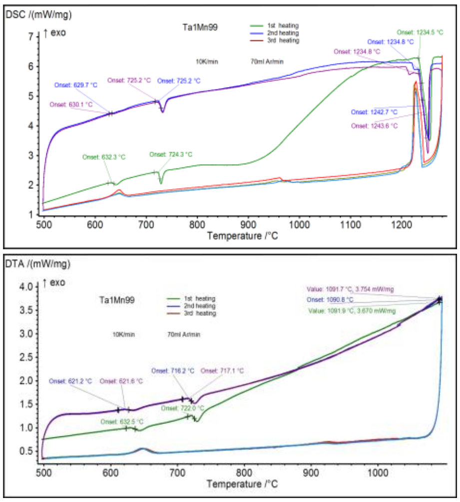

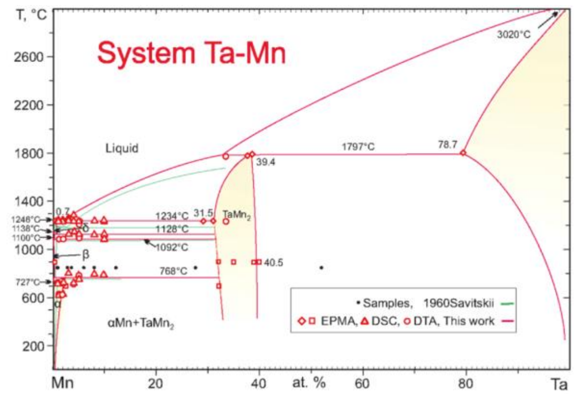

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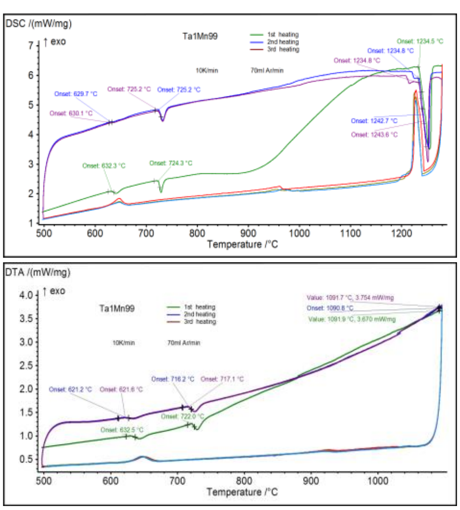

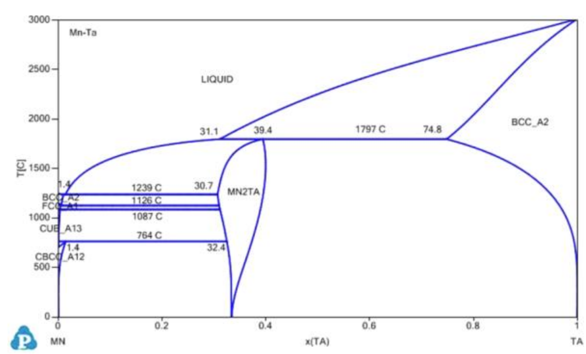

Source: X Yan *et al.*: Journal of Alloys and Compounds. 865(2021)158715.

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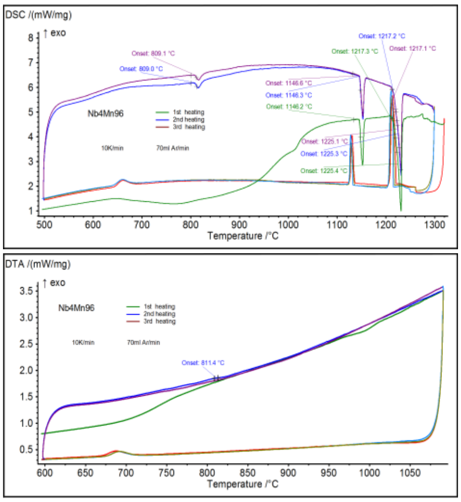
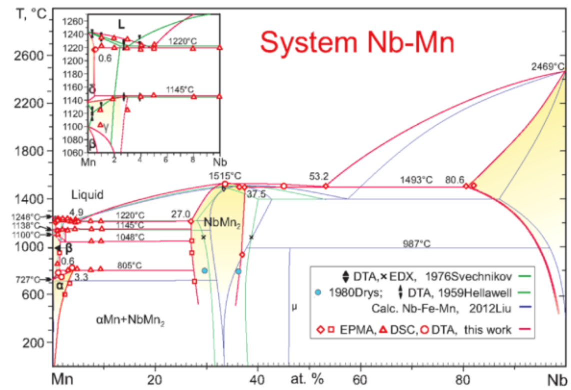
Example

Determination of phase diagrams

- Dynamic determination of Nb-Mn phase diagram using DTA/DSC (+ other thermochemical data)





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Determination of phase diagrams

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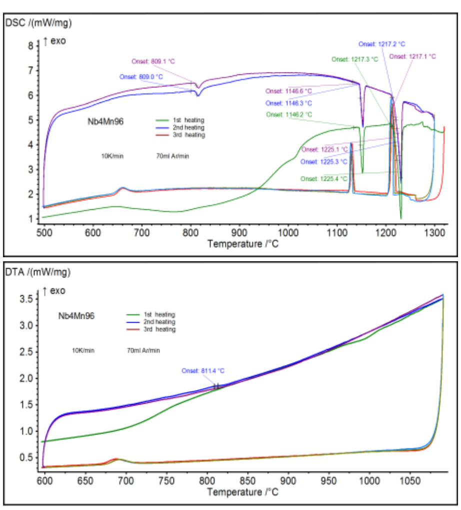

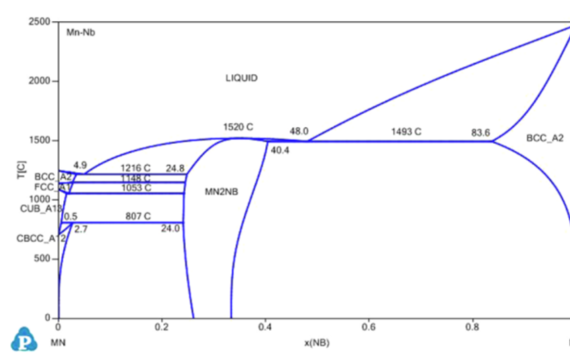

Source: X Yan *et al.*: Journal of Alloys and Compounds. 865(2021)158715.

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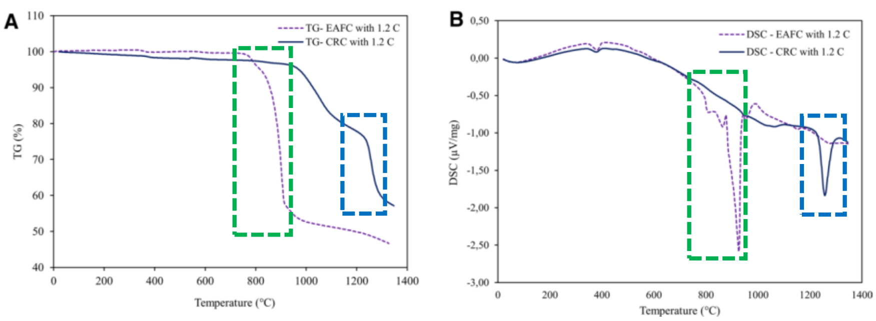
Example

Observation of material’s thermal behavior/stability using DSC with TGA

- Removal of volatiles
- Zinc removal from EAF and CRC dusts by heating as an example

Thermal analyses of CRC and EAF dusts: **a** TG and **b** DSC



Reduction and evaporation of zinc from EAF dusts

Reduction and evaporation of zinc from EAF dusts

Material	Chemical compositions of the dusts (wt%)								
	C	Fe_2O_3	ZnO	Cr_2O_3	CaO	MgO	MnO	SiO_2	K_2O
EAF dust	1.54	34.45	45.64	0.60	6.08	1.10	4.09	3.21	3.29
CRC dust	0.31	20.83	19.08	22.89	14.72	10.06	1.04	10.30	0.76

Source: M Omran, T Fabritius, Y Yu, E-P Heikkinen, G Chen & Y Kacar: Journal of Sustainable Metallurgy. 7(2021)1,15-26.

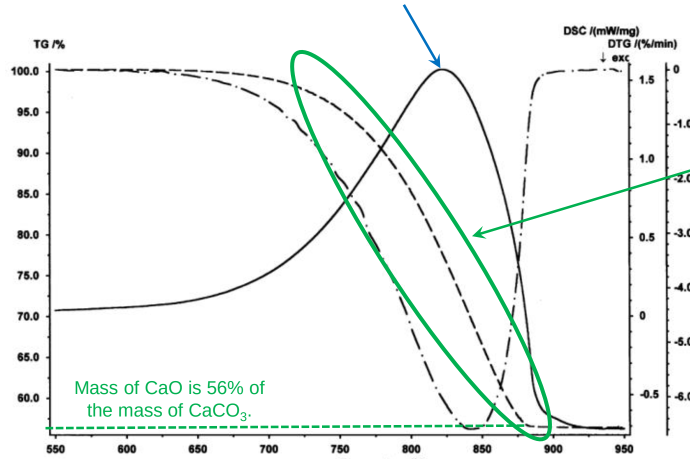
Example

Observation of material’s thermal behavior/stability using DSC with TGA

- Decomposition of compounds
- Decomposition of $CaCO_3$ during heating as an example

$$CaCO_{3(s)} \rightarrow CaO_{(s)} + CO_{2(g)}$$

Endothermic peak indicating the decomposition of carbonate.



Simultaneous decrease in mass corresponds with the removal of CO_2 from the sample.

Mass of CaO is 56% of the mass of $CaCO_3$.

Curves for the simultaneous TG/DSC of 30.5 mg of $CaCO_3$ heated at 16 °C min^{-1} in dry Ar: (—) DSC; (- - -) TG; (-·-·-) DTG.

Source:
J Sanders & P Gallagher: Thermochimica Acta. 388(2002)115-128.

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Example

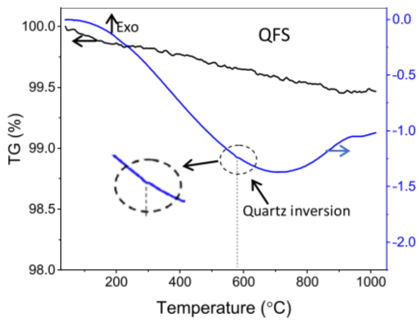
Observation of material's thermal behavior and characterisation of material

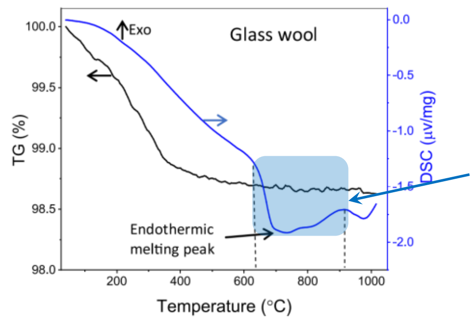
- Behavior of glass wool waste and mine tailings (quartz feldspar sand; QFS) during heating as an example

Chemical composition (wt%) of glass wool and QFS.

Samples	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	TiO_2	P_2O_5	MnO	SO_3	Other	SUM
QFS	77.5	13.5	0.2	0.3	0.0	4.8	3.3	0.0	0.1	0.0	0	–	99.9
Glass wool	63.4	1.9	1	8.3	2.5	16.1	0.6	0.0	0.0	–	0.2	6	100

TGA-DSC-curves of QFS and glass wool





Broad endothermic melting peak suggests that melting occurs on a broad temperature range. This could indicate the existence of amorphous material

Amorphous structure of the glass wool can be seen with e.g. XRD analysis (cf. lack of clear intensity peaks peaks which can be seen from GFS's XRD analysis).

Source: P Lemougna, J Yliniemi, H Nguyen, E Adesanya, P Tanskanen, P Kinnunen, J Röning & M Illikainen: Journal of Building Engineering. 31(2020)101383.

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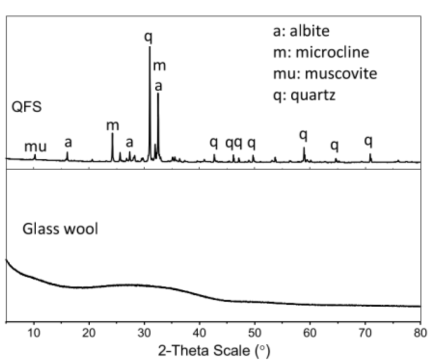
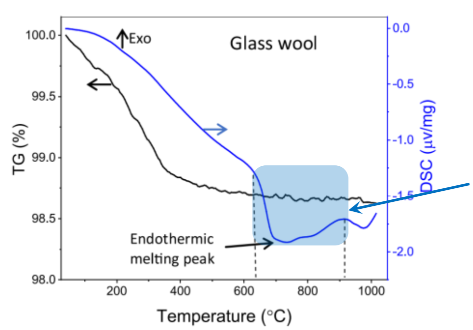
Glass wool	63.4	1.9	1	8.3	2.5	16.1	0.6	0.0	0.0	–	0.2	6	100

XRD patterns of QFS and glass wool





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Example

Determination of thermochemical properties

- Changes of enthalpy related to observed phenomena can be calculated from the DTA/DSC curves

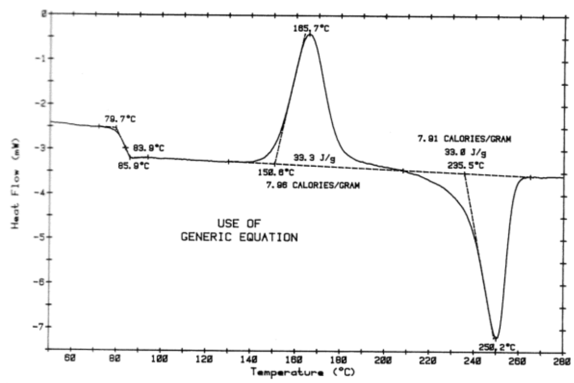
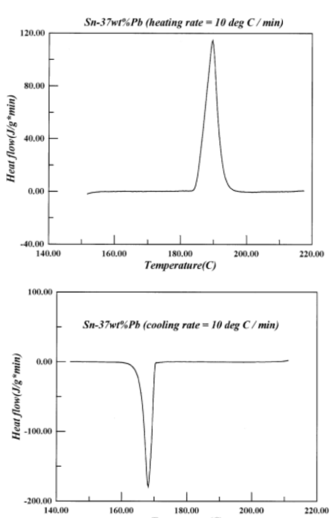


Figure from: Skoog & Leary: Principles of Instumental Analysis.

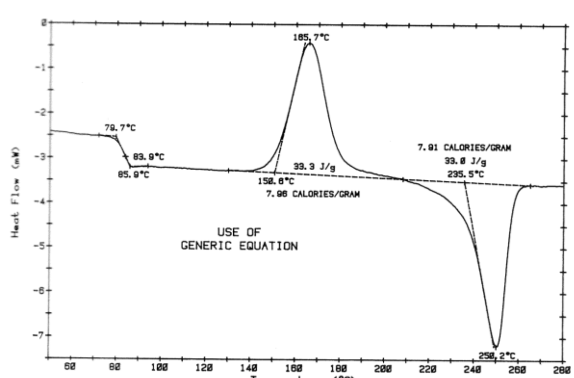
Example



Source: SW Chen, CC Lin & CM Chen: Metall. & Mater. Trans. A. 29A(1998)4,1965-1972.

Determination of thermochemical properties

- Changes of enthalpy related to observed phenomena can be calculated from the DTA/DSC curves
- Determination of enthalpies of fusion with DSC for various tin-based solder materials as an example



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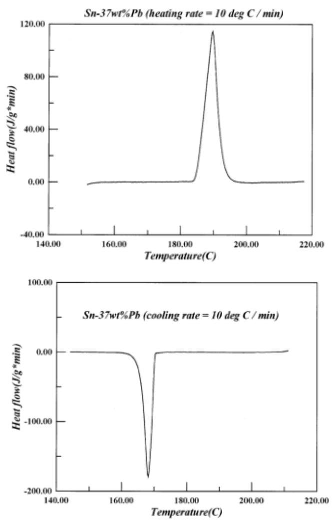
Example



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Determination of thermochemical properties

- Changes of enthalpy related to observed phenomena can be calculated from the DTA/DSC curves
- Determination of enthalpies of fusion with DSC for various tin-based solder materials as an example

Composition (Pct)	Scaning Rate (°C/min)	Reaction Temperature(s) (°C)			Enthalpy of Fusion (J/g)
Sn-3.5 wt pct Ag	10	222	—	—	68.2
Sn-3.5 wt pct Ag	−10	186	—	—	−65.6
Sn-3.5 wt pct Ag	1.25	218	—	—	66.4
Sn-3.5 wt pct Ag	−1.25	200	—	—	−64.6
Sn-58 wt pct Bi	10	139	—	—	43.2
Sn-58 wt pct Bi	−10	123	—	—	−42.7
Sn-58 wt pct Bi	1.25	138	—	—	49.1
Sn-58 wt pct Bi	−1.25	131	—	—	−49.8
Sn-37 wt pct Pb	10	183	—	—	45.2
Sn-37 wt pct Pb	−10	172	—	—	−44.
Sn-37 wt pct Pb	1.25	184	—	—	48.2
Sn-37 wt pct Pb	−1.25	172	—	—	−49.1
In-10 wt pct Ag	10	142	168	—	38.3
In-10 wt pct Ag	−10	233	136	—	−37.9
In-10 wt pct Ag	1.25	143	167	—	41.6
In-10 wt pct Ag	−1.25	240	140	—	−43.0
In-15 wt pct Ag	10	142	167	—	42.5
In-15 wt pct Ag	−10	279	136	—	−40.8
In-15 wt pct Ag	1.25	144	169	—	41.4
In-15 wt pct Ag	−1.25	288	140	—	−43.1
In-20 wt pct Ag	10	143	168	343	37.4
In-20 wt pct Ag	−10	323	136	—	−41.4
In-20 wt pct Ag	1.25	144	169	—	39.4
In-20 wt pct Ag	−1.25	329	143	—	−43.0
Sn-42 wt pct Pb-8 wt pct Bi	10	154	—	—	38.0
Sn-42 wt pct Pb-8 wt pct Bi	−10	171	162	128	−41.6
Sn-42 wt pct Pb-8 wt pct Bi	1.25	155	163	—	39.0
Sn-42 wt pct Pb-8 wt pct Bi	−1.25	179	171	—	−37.9
Sn-43 wt pct Pb-14 wt pct Bi	10	134	140	—	38.4
Sn-43 wt pct Pb-14 wt pct Bi	−10	160	151	128	−36.3
Sn-43 wt pct Pb-14 wt pct Bi	1.25	135	137	168	35.6
Sn-43 wt pct Pb-14 wt pct Bi	−1.25	166	159	134	−35.8
Sn-35 wt pct Pb-10 wt pct Bi	10	135	142	—	42.7
Sn-35 wt pct Pb-10 wt pct Bi	−10	147	142	128	−43.5
Sn-35 wt pct Pb-10 wt pct Bi	1.25	135	142	—	46.0
Sn-35 wt pct Pb-10 wt pct Bi	−1.25	164	134	—	−45.3
Sn-45 wt pct Pb-10 wt pct Bi	10	143	150	—	38.4
Sn-45 wt pct Pb-10 wt pct Bi	−10	176	160	130	−40.5
Sn-45 wt pct Pb-10 wt pct Bi	1.25	136	142	—	44.0
Sn-45 wt pct Pb-10 wt pct Bi	−1.25	179	162	—	−44.3
Sn-55 wt pct Pb-10 wt pct Bi	10	121	153	175	30.7
Sn-55 wt pct Pb-10 wt pct Bi	−10	197	153	128	−32.2
Sn-55 wt pct Pb-10 wt pct Bi	1.25	135	141	171	34.2
Sn-55 wt pct Pb-10 wt pct Bi	−1.25	202	163	134	−35.0

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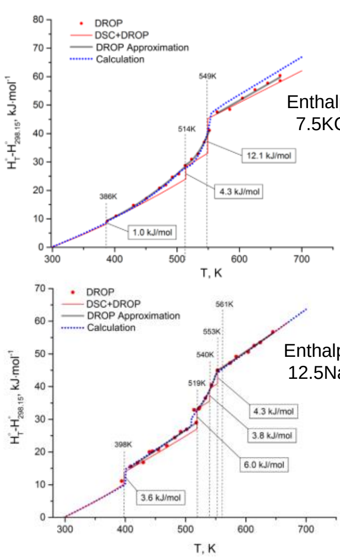
Example



Enthalpy changes for 7.5KCl-92.5$NaNO_3$

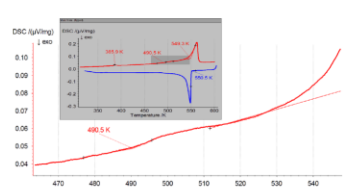
Enthalpy changes for 12.5NaCl-87.5KNO_3

Determination of thermochemical properties

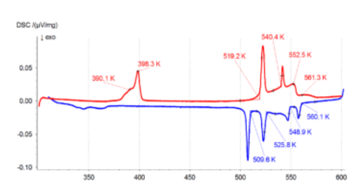
- Determination of enthalpies and heat capacities (C_P) of mixtures in the NaCl-KCl-$NaNO_3$-KNO_3 system as an example

$$C^{\circ}_{p(s)} = \frac{n_r}{n_s} \cdot \frac{DSC_s - DSC_b}{DSC_r - DSC_b} \cdot C^{\circ}_{p(r)}$$

- n = mole fraction of the substance
- DSC = signal from experiments (μV)
- Subscripts: s for sample
  r for reference (sapphire in these experiments)
  b for baseline



DSC curve for 7.5KCl-92.5$NaNO_3$ with a heating/cooling rate of 5 K/min



DSC curve for 12.5NaCl-87.5KNO_3 with a heating/cooling rate of 2 K/min

Source: D Sergeev, E Yazhenskikh, N Talukder, D Kobertz, K Hack & K Müller: CALPHAD. 53(2016)97-104.

Example

Determination of thermochemical properties

- Determination of enthalpies and heat capacities (C_P) of mixtures in the $NaCl-KCl-NaNO_3-KNO_3$ system as an example

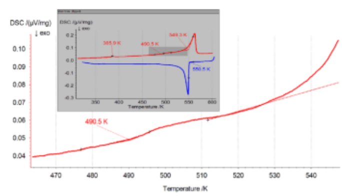
$$C^{\circ}_{p(s)}=\frac{n_r}{n_s}\cdot\frac{DSC_s-DSC_b}{DSC_r-DSC_b}\cdot C^{\circ}_{p(r)}$$

- n = mole fraction of the substance
- DSC = signal from experiments (μV)
- Subscripts: s for sample
  r for reference (sapphire in these experiments)
  b for baseline

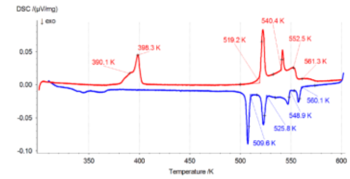
Coefficients of the linear equation of heat capacity (J mol[−1] K[−1]) $C^{\circ}_p = AT+B$ for the $7.5NaNO_3-92.5KCl$ and $12.5NaCl-87.5KNO_3$ mixtures taken from DSC measurements.

Temperature range (K)	A	± ΔA[a]	B	± ΔB[a]
7.5KCl–92.5 $NaNO_3$				
298–549	$2.241\cdot10^{-1}$	$5.81\cdot10^{-3}$	15.504	2.232
549–700	–	–	111.67	5.63
12.5NaCl–87.5KNO_3				
298–398	0.338	$2.80\cdot10^{-3}$	−15.258	−0.954
398–561	0.114	$4.29\cdot10^{-3}$	56.878	1.007
561–670	0.162	$2.24\cdot10^{-3}$	27.089	1.4

[a] Standard uncertainties are obtained taking into account the statistical scatter of the experimental data and are $u(C^{\circ}_p)=\Delta AT+\Delta B$ J mol[−1] K[−1].



DSC curve for $7.5KCl-92.5NaNO_3$ with a heating/cooling rate of 5 K/min



DSC curve for $12.5NaCl-87.5KNO_3$ with a heating/cooling rate of 2 K/min

Source: D Sergeev, E Yazhenskikh, N Talukder, D Kobertz, K Hack & K Müller: CALPHAD. 53(2016)97-104.

Summary

Differential Thermal Analysis (DTA) and Differential Scanning Calorimetry (DSC) are widely used in metallurgical R&D

- Possibility to study all the phenomena in which heat content of the sample changes
  - Removal of moisture/volatiles from materials
  - Decomposition of compounds
  - Reduction of materials
  - Oxidation of materials
  - Phase transformation (incl. phenomane in which mass does not change)
  - Characterisation of multicomponent systems
- Thermochemical properties of materials can be calculated based on results

Restrictions

- Equipment may limit heating/cooling rate, atmospheres, sample size, *etc.*
- Inaccuracies caused by the method must be known

DTA/DSC is often connected with other devices

- TGA, MS, sample analysis