Differential Scanning Calorimetry – Instruction Notes

Keywords: differential scanning calorimetry, phase transitions, enthalpy change, binary alloy, eutectic point

I. GOALS OF THE EXPERIMENT

Calorimetry measures the transfer of heat that occurs during temperature – induced processes in physical, chemical, and biological systems. Based on the measured quantities, conclusions can be drawn about the investigated thermodynamic system. Differential scanning calorimetry (DSC) is a technique in which the difference in heat flow rate to the sample and to the reference is recorded as function of temperature.

The purpose of this experiment is to get familiar with the power compensation DSC. In particular, an understanding of the thermodynamics of phase transitions in pure and mixed systems shall be achieved through analysis and interpretation of the measured quantities.

II. LEARNING CONTENT

- mode of operation and properties of a power compensation DSC instrument
- thermodynamics of phase transitions in unary and binary systems
- phase diagrams of binary systems
- thermodynamic properties of the eutectic alloys

III. PROCEDURE

- All samples should weigh between 6 and 10 mg.
- Use gloves while preparing the samples.
- Do not put the samples directly on the scale. You can use a piece of paper or measure directly in the sample holder.
- Use an empty cell as reference.
- Never touch the furnace lid with your fingers.
- Remove the furnace lid with tweezers and place it on the stainless steel plate.

FIRST LAB SESSION

1) **Measurements for calibration**

- Prepare an indium sample of ~ 8 mg.
- In ´*Setup*` → ´*Cell parameters*´ set τ_{lag} to zero, as this parameter is determined afterwards.
- Use the program *'Setup'* → *'Calibrate heatflow'* to determine the calorimetric sensitivity E(T) (~ 280 units/mW)
- Measure **the melting thermograms** of indium in the range 130 ℃ - 180 ℃ with four different heating rates rates β between 1 ℃/min and 20 ℃/min.

- Determine the onset-melting temperature for every thermogram using in the program *'Evaluation' → 'T onset/T endset'*
- Calculate τ_{lag} using the formula: $\tau_{lag} = (T_i^{onset} - T_j^{onset})/(\beta_i-\beta_j)$. Put this value in 'seconds' in *'Cell Parameters*'.

2) **Determination of melting and solidification temperatures of lead (Pb) and tin (Sn)**

- Prepare the samples: ~ 8 mg
- Record the **melting and solidification thermograms** of Pb and Sn at a scanning rate β = 10℃/min. Temperature ranges: 270°C – 370°C for Pb and 170°C – 270°C for Sn.
- Determine for both metals the melting and solidification temperatures. Keep in mind that these temperatures can differ significantly. Select the start and end temperature accordingly: $T^{Pb}_{melt} \approx 327$℃, $T^{Pb}_{solid} \approx 315$-324℃, $T^{Sn}_{melt} \approx 232$℃, $T^{Sn}_{solid} \approx 190$-210℃.
- Select manually the peak with a square area and with the command *'Evaluation' → 'Peak area'* determine the enthalpy change of both metals associated with the phase transition.

SECOND LAB SESSION

3) **Record the phase diagrams of different tin-lead alloys**

- Prepare alloys from tin and lead with the following Sn composition: 10%, 30%, 50%, 61-62% (eutectic point), 70%, 90%. Use the materials sparingly!
- The ceramic crucible should be free from sample rests before use. During mixing no oxidation of the metals should take place, therefore stir constantly during heating and remove the crucible directly from the flames after melting.
- Record for all 6 samples **the melting thermograms** in the range 150°C - 350°C with a scanning rate β = 10˚C/min. Each diagram, excepting that of the eutectic mixture, will show two melting peaks. **For the eutectic alloy record also the solidification curve**.
- Determine from the curves the onset temperatures of the phase transitions (for both peaks if possible) and the enthalpy changes.
- Compare all results with the literature data.

IV. REFERENCES:

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