

Topic: Design and Operation of a Nano-Calorimeter for Highly Sensitive and Fast Heating Rates Measurements

A thin-film differential scanning calorimeter TDSC is a device by which to investigate the themo-physical properties of thin films on the nanometer scale. TDSC can be used in investigation of the melting properties of small (down to 2 nm diameter) particles, glass transition and crystallization processes. By using the TDSC technique, it is possible to approach adiabatic working conditions, since the extremely fast heating rate provides no time for dissipation of the heat from the cell to its surroundings. In this case all power generated by the heater is consumed by increasing the temperature of the cell.

The remarkable low thickness of the cell constituents, i.e. the heater and the membrane, provides extremely small addenda, which is the key to the high sensitivity of the TDSC method. The heat pulse technique allows determining the dynamic behaviour of the calorimeter system as well as that of the sample. From the pulse response the heat transport properties can be derived and the measured signals can be corrected properly so that quantitative results for the heat capacity of the sample can be obtained at every moment of the measurement. The disadvantage of such a method is that every new sample needs a new calorimeter chip. Using electrical thermal annealing technique we can get ultrafast heating rates, up to 10⁴ K/s, via a direct resistance heating. TDSC measurement is initiated by supplying a short, 10 msec dc current pulses to the Pt strip, thus raising the temperature of the system by joule heating. The voltage and current are monitored in real time during the pulse and thus the power supplied to the quasi-adiabatic system is obtained directly.

At these fast heating rates the temperature of the micro-heater system is determined by measuring the changes in resistance of the metal strip. In order to evaluate the data in terms of standard thermodynamic parameters the raw data are evaluated in terms of the heat capacity Cp. All measurements will be performed in a vacuum chamber under high vacuum conditions.