Multiparallel Power Compensated Calorimetry for Ultrasmall Samples

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Conventional differential scanning calorimeters are typically limited to the analysis of macroscopic samples in a one at a time approach. However, the rational design of new materials with tailored properties may greatly benefit from high-throughput screening techniques. In this presentation we show a first attempt towards a rapid-screening calorimetric tool.

We have designed and developed a multiparallel sensitive scanning calorimeter for use with microgram or submicrogram samples in the temperature range from 77-700 K. This development is based in our previous work with single calorimeters [1]. Semiconductor processing techniques are used to fabricate 64 microreactor-based calorimeters with an individual active area of 4×4 mm² on a 4” wafer. The small heat capacity of the addenda, around 150 nJ/K at room temperature, allows for scanning rates up to 100 K/s in power compensation mode. A software-based digital proportional integral controller was developed using LABVIEW to accurately maintain the predefined setpoint (heating ramp) by adjusting the current outputs. A Field-Programmable Gate Array Card (NI) is used to process eighth I/O signals simultaneously. A custom-made electronic circuit amplifies and converts the voltage output of the card to a current used to heat the Pt resistance of the microreactors. A multiplexed system integrated in the electronic circuit allow for rapid switching between the different elements. The use of the multiparallel calorimeter and its dynamic sensitivity are demonstrated by analyzing the melting behavior of In thin films and the interface reaction occurring in Mg/Cu films of different stoichiometry.