Chapter 2

Fast Scanning Chip Calorimetry

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2.1 INTRODUCTION

Fast scanning chip calorimetry (FSC) has recently been introduced as an analytical tool for calorimetry-based thermal analysis of materials at conditions which cannot be achieved by conventional differential scanning calorimetry (DSC) [1]. Historically, the development of chip calorimeters was driven by the goal to analyze smallest amount of samples or thin films, with first reports published in the mid-1990s [2,3]. According to the basics of scanning calorimetry [4–7], measurement of small heat capacities requires application of high scanning rates since the measured signal (heat flow rate) is proportional to heat capacity and scanning rate. The initial devices were operated under quasi-adiabatic conditions, allowing measurements on heating only. Heating rates of 30,000 K/s were sufficient for thermal analysis of Sn films with a thickness as low as 5 nm [3]. At this time the high scanning rates were used mainly as a means for the analysis of thin films and not for evaluating the kinetics of thermal transitions. Extreme high heating rates of bulk samples were commonly realized by heating the sample by either a current through the wire-shaped sample (up to 10^8 K/s) [8] or by laser heating [9]. These techniques which do not allow for controlled cooling of the sample will not be further considered here.

In the case of polymer-related research the importance of fast scanning devices for possible adjustment of specific states of structure was well recognized at that time [10–15]. A main, initial driving force was the aim to mimic polymer solidification in industrial processes, which may involve cooling rates of the order of magnitude up to 1000 K/s [16–20], or to avoid reorganization of structure during heating, needed to obtain information about the thermal stability of structures evident before analysis. These motivations then led to the development and application of modified DSC instruments such as

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