



Differential Scanning Calorimeter

Thermal analysis of plastic bottles: PET and PE



Chip-DSC 1

Introduction

Thermal analysis is a very useful tool for the analysis of various compounds. Differential Scanning Calorimetry (DSC) gives information about phase changes and chemical reactions.

Plastic bottles consist of two parts: the transparent body and the opaque cap. In this application note, we show how to identify them and show some characteristic behavior of plastics in DSC-analysis.

Methods

Using a DSC for analysing materials is a common technique. In this application, the Chip-DSC was used for measurements of the polymers of a plastic bottle. The transparent body and the opaque cap were analysed separately and identified as polyethylene (PE) and polyethylene terephthalate (PET).

While for PE only the endothermic melting point around 130°C can be observed, PET shows a glass transition followed by a cold crystallization before the material melts.

The DSC signal in general is generated by heating a pan containing a sample and an empty reference pan using the same heat source. Subtracting the heat flow signals of the two pans from each other, results in endothermic or exothermic peaks if the sample temperature changes due to thermal effects. In case of amorphous materials the so called glass transition can be seen as a change in specific heat capacity (C_p).

The Chip-DSC 1 integrates all essential parts of a DSC, furnace, sensor and electronics, in a miniaturised housing. The chip-arrangement comprises the heater and temperature sensor in a chemically inert ceramic arrangement with a metallic heater and a temperature sensor. Therefore, the Chip-DSC allows a very fast heating and cooling speed combined with high resolution and accuracy as well as reproducibility.

Table 1. Experimental Conditions

Instrument	Chip-DSC 1
Heating rate	50 K/minute
Sample Mass	approx. 10 mg
Sample Pan	open aluminum pans without lid
Gas	Static air

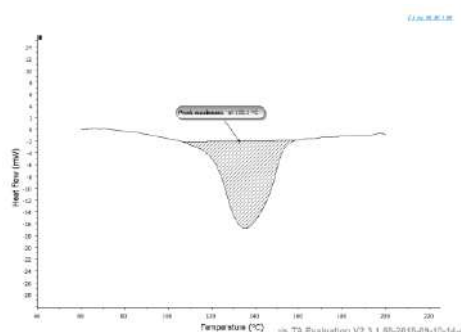


Fig 1: cap; second heating; heating rate 50 K/min

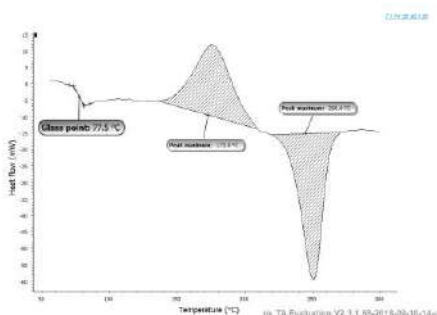


Fig 2: body; second heating; heating rate 50 K/min

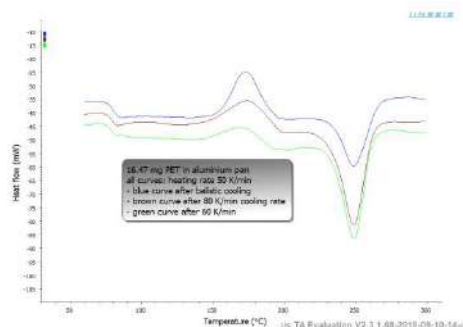


Fig 3: PET; second heating after cooling at different cooling rates (quench cooling, 80 K/min and 60 K/min)

Experimental

Small pieces were cut from the bottle (cap and body) and weight in a crucible. The samples were run on a heat flux Chip-DSC 1. After a first heating run followed by rapid cooling, the samples were run a second time under the conditions shown in Table 1.

Results

Figure 1 shows the DSC profile of the cap. Melting occurs at 135°C which is a typical value for PE. Melting points from different caps show slightly varying melting points in the range of 125 to 135°C indicating different chain lengths.

Figure 2 shows the result for the second heating of the transparent part. The glass transition can be seen at 77°C followed by an exothermic cold crystallisation at 176°C and the endothermic melting at 250°C. This is a typical behavior for PET.

Figure 3 shows the second heating after a controlled cooling with different rates (quench cooling, 80 K/min and 60 K/min). Depending on the previous cooling rate, glass transition and cold crystallisation change in aspect : the faster the previous cooling the better these phase transitions can be seen. At low cooling speed the polymer chains have enough time to crystallize.

Separation of these to polymers for recycling is done in the following way:

After shredding the compressed bottles with their caps, the resulting granulas are separated by floating in water. PET having a higher density than water will sink while PE swims.