



Differential Scanning Calorimeter

Thermal analysis of Polymers: Oxidation induction time (OIT)



Chip-DSC 10

Introduction

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

Especially in the field of polymer analysis, the DSC technique is a very important and frequently used method to determine critical parameters and specific values that characterizes the material. One of these values is the oxidation behavior and aging behavior which can be checked using the OIT test.

Methods

Information about the stability of materials and especially polymers can be obtained from the analysis of decompositions reactions. One widely used standard test method is the measurement of the *oxidation induction time* (OIT). The differences in stability toward oxidation between materials can be clearly seen here. The use of such measurements allows also thermally, mechanically or chemically stressed material to be distinguished from fresh material.

The oxidation behavior can be investigated by DSC in two different modes:

- The OIT (oxidation induction time) is determined in a run with an isothermal segment where the atmosphere is changed from inert to oxygen. Then the time between the change of atmosphere and the onset reaction of the oxidation is measured at several isothermal segments to give the oxidation induction time at a specific temperature.
- The OOT (oxidation onset temperature) is the simpler but less common method, where a constant heating rate is used in oxygen atmosphere. Here, the temperature when the sample reacts is just monitored in the linear heating segment and gives the OOT directly.

Table 1. Experimental Conditions

Instrument	Chip-DSC 10
Heating rate	10 K/minute
Sample Mass	approx. 10 mg
Sample Pan	open aluminum pans without lid
Gas	Argon / Oxygen flow of 10 l/h

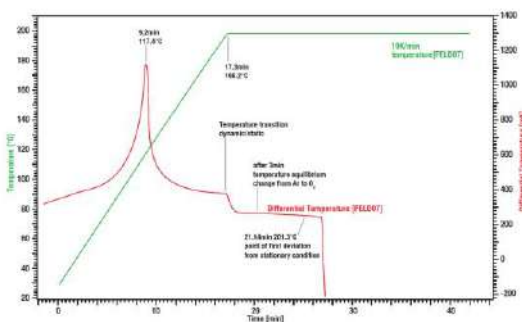


Fig 1: OIT experiment with LDPE sample. 10K/min RT to 200°C, isothermal segment at 200°C with switch from AR to O₂

Experimental

A small piece of LDPE granules was put into an aluminum crucible and placed on the Chip Sensor of a Chip-DSC 10. The system was evacuated and filled with argon atmosphere. The sample was then heated with 10K/min at constant argon flow rate of 10 l/h. At 200°C, an isothermal segment was applied and the gas flow was switched from argon to oxygen at 10 l/h.

Results

Figure 1 shows the DSC profile of the experiment vs. Time. At the point where the heating ramp goes into the isothermal segment, there is a response « step » in the heatflow signal. After the signal has become stable again, the atmosphere was changed to oxygen. Seven minutes after that, the heat flow shows a significant change, which symbolizes the onset of the oxidation. The time between the change of atmosphere and the onset of the oxidation can be determined and gives the OIT time at 200°C for this sample, which is around 7 minutes.

This experiment can be done at other temperatures, getting closer to the melting point of the substance. Out of the change of the OIT at several isothermal temperatures, a good prediction of aging and thermal stability of the polymer could be obtained. Experiments like this one are typical applications for DSC in polymer fields and crucial for the characterization of polymer containing materials.