

Oxidative Stability of Polymers: The OIT Test

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Introduction

Degradation of a polymer goes hand in hand with worsening of its properties and shortening its lifetime, so that stabilizers are usually added to avoid oxidative decomposition or to slow it down. An easy and quick method of measuring the stability of polymers against oxidative stress is to perform oxidation induction tests, also called OIT, with a differential scanning calorimeter (DSC). These tests are internationally recognized and described in different standards, e.g., ASTM D3895-92, ASTM D6186, EN 728 and ISO 11357-6.

Measurement Conditions

In the following, OIT tests are carried out on two different polyethylene samples using the DSC 300 *Caliris*[®] *Classic*. In order to ensure good repeatability of the DSC curves, the samples were prepared exactly the same way

and the measurements performed with crucibles without lid. In fact, this type of test is very sensitive to sample preparation and measurement parameters, and even the hole size in the lid would influence the results.

The test consists of three different steps:

- Heating to a temperature higher than the melting temperature of the sample under a dynamic nitrogen flow
- Isothermal segment for 3 minutes under nitrogen
- Switching the gas from nitrogen to synthetic air until degradation occurs. The end of the test is automatically detected via the *Proteus*[®] measurement and evaluation software

Table 1 summarizes the measurement conditions.

Table 1 Conditions of the OIT test

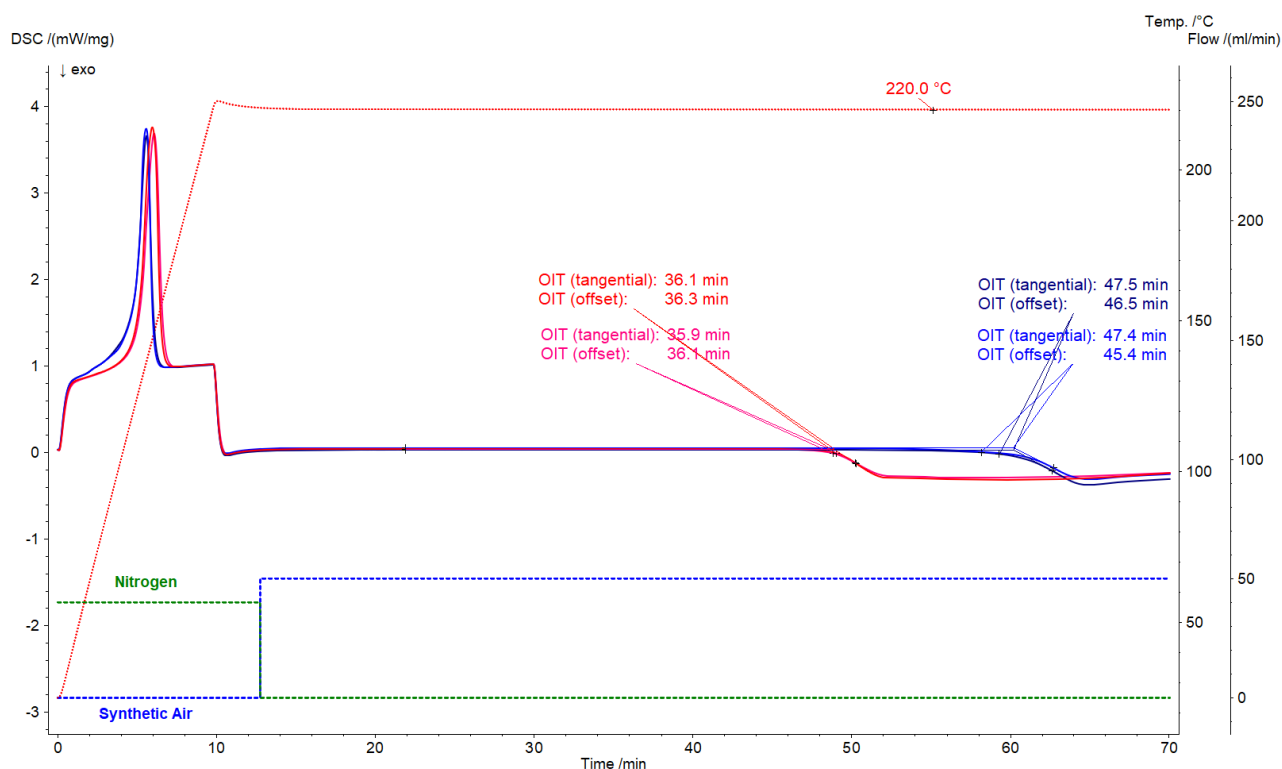
Device	DSC 300 <i>Caliris</i> [®] <i>Classic</i>
Crucible	<i>Concavus</i> [®] (aluminum), open
Sample mass	10.2 ± 0.2 mg
Temperature program	25... 220°C, 20 K/min, nitrogen (40 ml/min) 220°C, 3 min, nitrogen (40 ml/min) 220°C, synthetic air (50 ml/min)

Test Results

Figure 1 depicts the test results. The endothermic peak detected during heating is due to melting of the polyethylene. Oxidative degradation is characterized by an exothermic effect during the isothermal segment. Here, it takes place 35.9 to 36.1 min (tangential evaluation) after switching the gas to synthetic air for sample 1 (red and pink curves). Sample 2 exhibits a better oxidative stability with an OIT value of more than 10 min higher (blue and dark blue curves). The curves show the excellent repeatability of the measurements.

Conclusion

An OIT test allows for easy and fast characterization of the oxidative stability of polymers and comparison of their thermo-oxidative performance. Careful sample preparation combined with the outstanding measurement performance of the DSC 300 *Caliris® Classic* and the automatic evaluation feature of *Proteus®* ensure good repeatability of the results.



1 OIT measurements on 2 polyethylene samples