



APPLICATION NOTE

Thermal Analysis

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How to Optimize OIT Tests?

Introduction

OIT is the abbreviation for oxidative induction time. It provides information about the oxidative stability of mainly polymer materials. This information is important because plastic parts age throughout their lifetime due to exposure to environmental elements such as heat, oxygen, light and radiation. Aging causes the degradation of the physical properties of the polymers and will lead to their failure. Antioxidants are often added during the plastics formulation to hinder the aging caused by oxygen and to increase their lifetime. Because the reaction between polymers and oxygen (O_2) is exothermic, OIT determination using Differential Scanning Calorimetry (DSC) is an ideal solution to study this process.

In the OIT test method, the test specimen is heated to a specific temperature in an open pan under an inert atmosphere. After a short period of time, at the isothermal temperature, the gas is switched from inert purge to O_2 , or air purge. The time taken from the O_2 gas switching to the onset of oxidation reaction is defined as OIT. OIT is an accelerated test used as a qualitative evaluation of the oxidative stability of a material. This time can be used as criteria of the thermal stability of polyolefins in an oxidative atmosphere. The OIT method is standardized in ASTM® D 3895 and DIN EN 728 for polyolefins. The OIT test is of great interest to companies who produce or utilize polyolefins, which are used at elevated temperatures under an oxygen atmosphere such as tubes or pipelines, cable or wire insulation, geomembranes or vapor-barrier-films. It can be used to compare the aging resistance of different plastics, to detect the impact of antioxidant concentration and to assess the effectiveness of antioxidants.

In this application note, we will review several influential factors for the OIT method. They are:

- Isothermal measurement temperature
- Sample selection
- Sample geometry
- Pan material

The data was generated on the PerkinElmer® Jade DSC. The Jade DSC has been designed to be both rugged and reliable. The system utilizes a precisely-machined disc of hardened nickel chromium and a low-mass aluminum coated furnace to give the instrument high resistance against contamination or aggressive gases, and to provide accurate, reproducible results. The integrated gas flow control and switching device assures high stability and best control over purge gases.

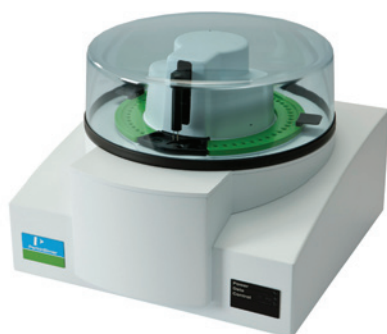


Figure 1. DSC 6000.

Experiments and discussions

Isothermal temperature

The crucial parameter in OIT determination is the isothermal temperature. This temperature is usually above the melting temperature for crystalline polymers. Low temperature will make the experiment too long and may not be acceptable economically, while high temperature can cause fast oxidation and lead to poor reproducibility and resolution. As shown in Figure 2, increasing the isothermal temperature from 205 °C to 215 °C resulted in decreased OIT. Usually an OIT between 5 and 60 minutes is desired. So in this case, isothermal measurements at 205 °C are the preferred choice.

Sample selection

For sample selection, the storage time (the time interval between when the part is made and when the actual OIT measurement takes place) should be taken into consideration because the oxidation starts as soon as the plastic part is manufactured. The antioxidant additive may be consumed during processing and storage, which can lead to a lower OIT compared with a virgin part. The sampling

location on the part also has an impact on the OIT result because various locations of the sample may have different concentrations of antioxidant additives. For example, in the case of a water pipe, the sample taken from the outside of the pipe may have a lower OIT than the sample taken from the center of the pipe because the antioxidant additive at the surface can be extracted by water.

Sample geometry

Sample geometry is another important influential factor in OIT measurements. Since oxidation happens between sample and oxygen, the surface area relative to the sample volume is important. Different sample geometries have different surface-to-volume ratios. Figure 3 illustrates the effect of sample geometry on the onset time and rate of oxidation. In this example, the standard sample was a compact disk.

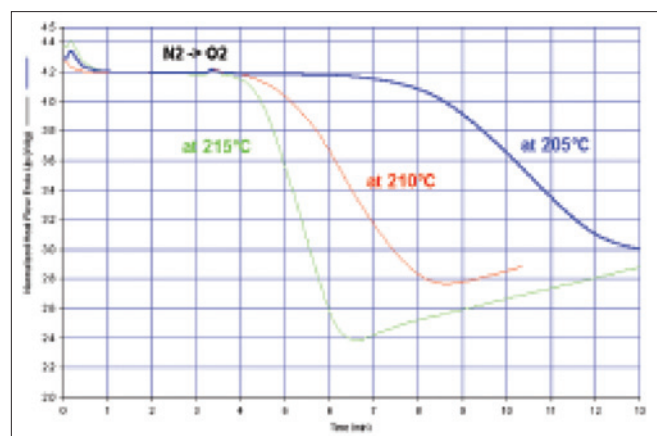


Figure 2. Influence of isothermal temperature on OIT for LLDPE.

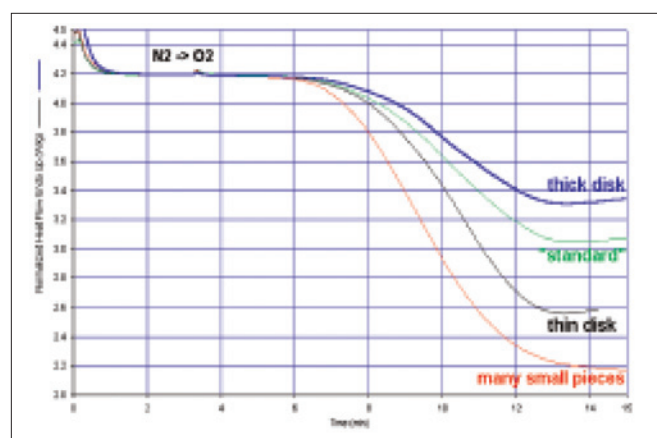


Figure 3. The effect of sample geometry on OIT measurement of LLDPE at 205 °C.

Pan material

The graph shows the normalized heat flow rate over time for two different pan materials. The y-axis represents the normalized heat flow rate in W/kg, ranging from 3.0 to 4.5. The x-axis represents time in minutes, ranging from 0 to 18. Two data series are plotted: 'N2 → O2' (blue line) and 'Aluminium Pan' (red line). The 'N2 → O2' series starts at approximately 4.45 W/kg, remains relatively constant until about 3.5 minutes, then drops sharply to a minimum of about 3.1 W/kg at 9 minutes, before rising slightly to 3.3 W/kg at 10 minutes. The 'Aluminium Pan' series starts at approximately 4.45 W/kg, remains relatively constant until about 8 minutes, then decreases steadily to about 3.1 W/kg at 12 minutes, and remains constant thereafter.

Time (min)	N2 → O2 (W/kg)	Aluminium Pan (W/kg)
0	4.45	4.45
2	4.45	4.45
4	4.45	4.45
6	4.45	4.45
8	4.45	4.45
10	3.3	4.2
12	-	3.1
14	-	3.1
16	-	3.1
18	-	3.1

Figure 4. The influence of sample pan material.

Data analysis

In the calculation window you can find the analysis for OIT (Figure 5). The standard package of the PerkinElmer Pyris software also includes the Pyris Player. The Player software is useful in many cases. It allows you to automate the work routine. Not only can this software be used with an autosampler, this package offers the user to option to set up a sequence that will automatically calculate the OIT at the end of a single sample run.

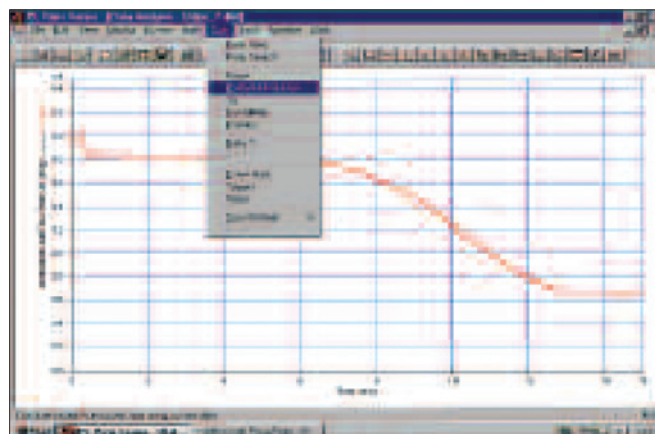


Figure 5. The OIT calculation window in the Pyris software.

Another valuable feature is the tolerance test. The customer determines the criteria for pass or failure of the test. The OIT criterion can be within a range, greater than or less than a defined value. When using an autosampler the customer can specify the action upon failure. The action can be to either stop, continue, skip to the next sample or block the sequence.

One final example of OIT application is given in Figure 6. It is the measurement of a HDPE pipe piece. The temperature was ramped from 30 °C to 200 °C at 20 °C/minute and held isothermally. The gas switched from N₂ to O₂ at 2 minutes on the isothermal hold. The onset, at about 95 minutes, defines the time interval from time zero (switch to O₂) to the onset of the oxidation reaction. The tolerance test feature confirms that this material passed the test.

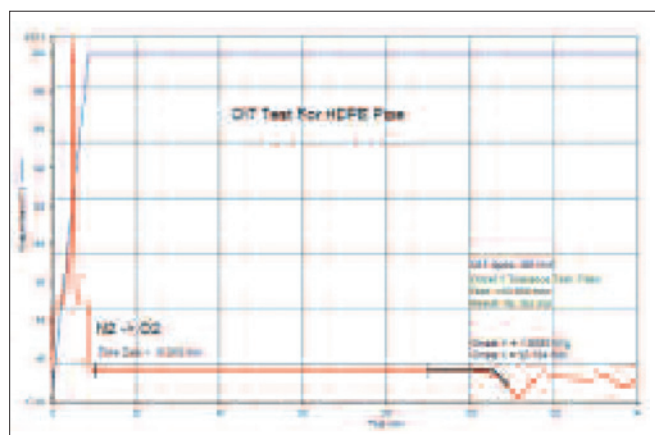


Figure 6. OIT test for HDPE pipe.

Summary

In order to get good, reproducible OIT data, you need to assure a:

- Stable isothermal temperature
- Constant sample morphology, geometry and weight
- Reliable and consistent purge gas flow rate
- Similar pan material is used

The Jade DSC with integrated mass flow controller and Pyris software is a great combination to perform OIT tests. It is an ideal, cost-effective solution for reliable tests in QA/QC labs or as part of product development.

Reference

Gottfried W. Ehrenstein, Gabriela Riedel and Pia Trawiel, Thermal Analysis of Plastics, 2004.