APPLICATION NOTE

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NEXTA DSC

Oxidation Induction time (OIT) measurements

INTRODUCTION

Oxidation Induction time (OIT) is an oxidation acceleration test that is used to predict a polymer's stability when in normal use. A standard procedure used in production, OIT is also used in R&D environments to evaluate the effectiveness of additives within the polymer for reducing oxidation.

The oxidation of polymers, such as polyethylene, in the atmosphere adversely affects the performance of the polymer component. The properties that are crucial to the part's effectiveness, such as mechanical strength or electrical insulation, can deteriorate significantly once the part begins to oxidize. Polymer manufacturers overcome this issue by adding antioxidant agents to the polymers. OIT is a useful technique that evaluates the effectiveness of these additives, giving manufacturers and researchers a fast method of evaluating long-term polymer stability.

During an OIT test, a small amount of polymer is heated in an inert atmosphere to above melting point, at which point oxygen is introduced into the system. The sample is held at a constant temperature within the oxygen environment until the polymer begins to react (decomposition). The typical temperature range of the oxygen phase is between 190ºC and 220ºC, or as much as is needed for the polymer to melt. The time taken from melting to decomposition within this test is measured and is used to predict polymer behavior at room temperature and standard atmospheric conditions.

Differential scanning calorimetry (DSC) is an established method for measuring OIT as the temperature can be controlled precisely, and the decomposition point of the sample can be easily detected. Standard test methods for measuring OIT, such as ASTM® D 3895, ISO 11357-6:2018 and DIN EN 728 for polyolefins, give specific instructions for DSC operation. The NEXTA DSC by Hitachi High-Tech is ideal for OIT measurements due to its unique furnace design and operates well within the tolerances given by the standard test methods.

Hitachi High-Tech Analytical Science's family of thermal analyzers have been employed in the field for more than 45 years, delivering world-class performance for precise materials and process characterization measurements, such as OIT measurements of polymers.

HITACHI INSTRUMENTS FOR POLYMER OIT MEASUREMENTS

NEXTA DSC

The NEXTA DSC200 is a high-performance DSC instrument, designed to be easy to use and meet the requirements of test methods ASTM D 3895 and DIN EN 728. High sensitivity and world-class baseline performance enable the analyzer to easily measure the time between melting and decomposition. The NEXTA DSC200 is also an extremely versatile instrument, with options for automated operation, and can be expanded post-installation for new applications.

For oxidation induction time determination, the unique furnace design of the NEXTA DSC200 delivers excellent baseline stability for holding the furnace at a constant temperature and getting a reliable result. The advanced gas controller unit allows for automated, rapid and efficient changeover from the nitrogen to oxygen atmospheres, reducing wait time and increasing throughput.

In addition to low noise and excellent sensitivity of the NEXTA DSC200, the instrument is very low cost to run, with few consumables needed. The robust design also ensures minimal downtime, supporting fast-paced polymer characterization.

The auto-analysis function, together with the auto-sampler for up to 50 samples, helps to speed up analysis time and free up operators. And the unique RealView camera system allows you to watch the analysis in real time, which can be invaluable for troubleshooting or the development of new materials.

SOFTWARE GUIDANCE FUNCTION FOR EASY ADHERENCE TO STANDARD TEST METHODS

The NEXTA DSC range comes with Hitachi's intuitive and advanced NEXTA TA software, which gives you a choice in how to operate the instrument. New users are able to get reliable and accurate results when measuring to standard test methods by using the guidance mode. This mode walks users through the steps necessary to make a measurement that complies with standard test methods and includes procedures for both ASTM and ISO.

More experienced users can use the software in standard mode for more advanced analysis.

PERFORMANCE AND RESULTS

The NEXTA DSC200 was used to evaluate the OIT of polyethylene (PE) under the following conditions:

Sample weight: 5mg
Atmosphere: Nitrogen at 40mL / min
Oxygen at 40mL / min
Isothermal temperature: 200ºC, 205ºC, 210ºC and 215ºC (all above melting point and the temperature is set by the NEXTA TA software).

Oxidation induction time measurement result for PE at 205ºC

Figure 1 shows the DSC curve after the PE has melted and the atmosphere has been changed from nitrogen to oxygen. The exothermic part of the curve at just after 17 minutes indicates the onset of oxidation. The OIT time of PE at 205ºC is measured as 17.2 minutes.

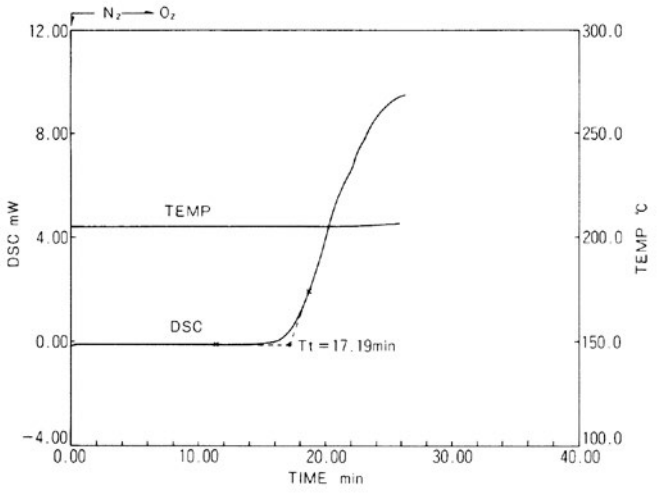

Figure 1 OIT measurement result for PE at 205ºC

Temperature dependence of oxidation induction time for PE

Figure 2 shows the effect of temperature on OIT. The higher the isothermal temperature, the shorter the oxidation induction time.

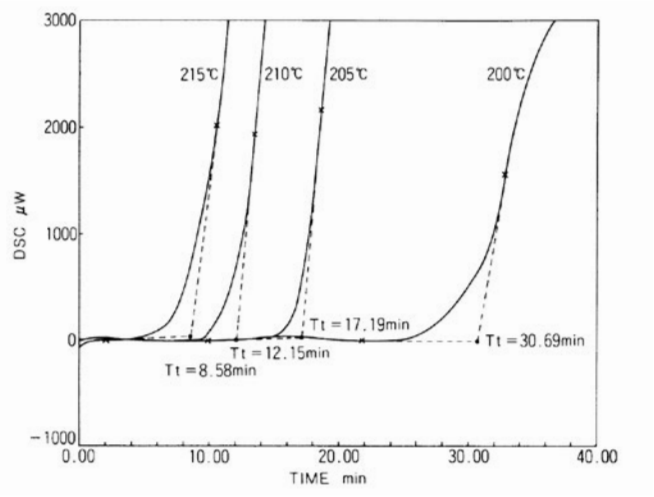

Figure 2 Temperature dependence of OIT for PE

The effect of copper on OIT of PE

Figure 3 shows OIT for a sample of PE that contains copper. OIT is only six minutes in this case, much shorter than the 17 minutes seen in figure 1 above. We can conclude that Cu accelerates the oxidation of PE. This type of analysis is useful for evaluating the oxidative effects of new additives. This experiment can be carried out using the Copper or Aluminum pans that are available to go with our TA instruments.

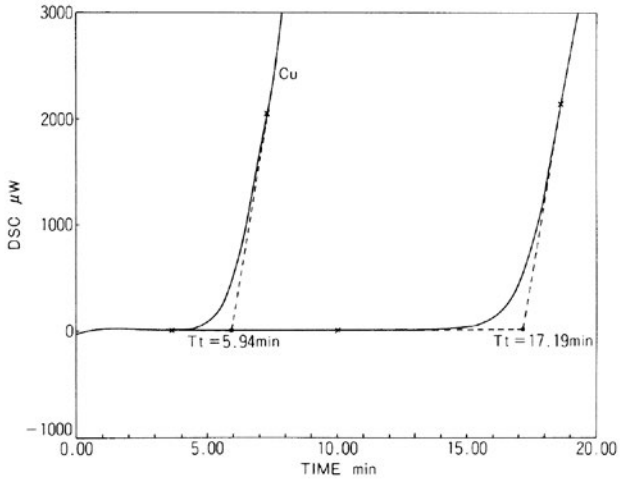

Figure 3 The effect of Cu on OIT for PE at 205ºC

OIT for different grades of PE

In figure 4, three different grades of PE were tested. You can see that the OIT range is from 4.25 to 17.19 minutes, meaning the stability of different types of PE vary enormously.

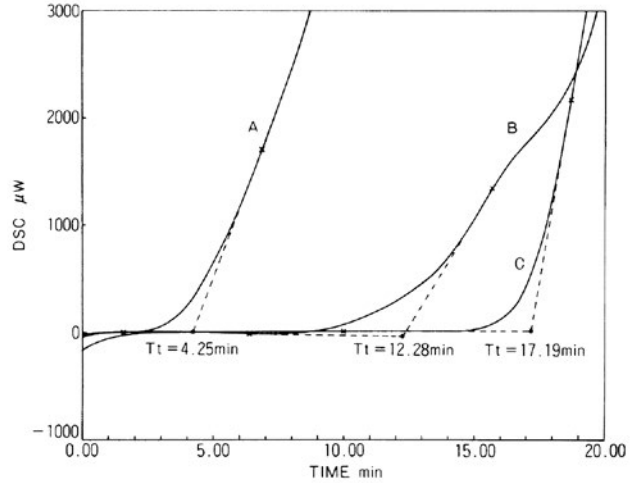

Figure 4 OIT for three different PE grades at 205ºC

SUMMARY

The **NEXTA DSC200** reliably and accurately determines the oxidation induction time of polymers. Excellent baseline performance and high sensitivity ensures the measurements fall well within the accuracy demanded by the standard test methods for this procedure. Simple to use and robust, the NEXTA DSC range includes a guidance function, walking operators through the tests in accordance with the standard test methods, ensuring the oxidative stability of manufactured polymers is correctly established.

Many applications have been optimized for Hitachi High-Tech Analytical Science's thermal analyzers. For more information on other applications, please contact our experts at **contact@hitachi-hightech.com**.

NEXTA DSC SERIES: HIGH ACCURACY MATERIALS CHARACTERIZATION

Designed for accurate determination melting point, glass transition and crystallization temperatures, our range of differential scanning calorimeters deliver excellent sensitivity and baseline flatness.

The NEXTA DSC range offers:

- High sensitivity and baseline performance, with unique furnace design for accuracy Real View camera system that allows you to watch material behavior on screen
- Intuitive, easy-to-use software, with advanced functionality for specific applications
- Reliable auto-sampler testing and auto analysis function for faster testing
- High degree of flexibility, allowing for addition of options after installation

Visit **www.hitachi-hightech.com/hha** for more information.