SII NanoTechnology Inc.

OXIDATIVE INDUCTION TIME (OIT) MEASUREMENTS BY DSC

Oxidative induction time (OIT) measurements provide a valuable characterization parameter associated with the long-term stabilities of polyolefin materials, particularly polyethylene. Cables utilized for telecommunication purposes are generally encased in a coating comprised of polyethylene. In order to ensure that the cable coatings will exhibit an acceptable level of long-term stability, a test needs to be performed which will provide a relative and reliable indication of the stability of the polyethylene coating.

Differential scanning calorimetry (DSC) provides an easy to use, and sensitive means of characterizing the thermal properties of polyethylene materials. The technique measures the heat flow into or out of a sample as the sample is either heated, cooled or held isothermally. For polyolefin materials, DSC can be used to measure the following major quantities:

- Melting points
- Heat of melting
- Percent crystallinities
- Glass transition temperatures
- Crystallization times and temperatures
- Thermal stabilities
- Oxidative stabilities

The Seiko EXSTAR DSC6200 provides excellent, sensitive and reproducible results, including oxidative induction time measurements, on polyolefin materials. The instrument offers the following desirable features:

- Outstanding baseline performance
- High sensitivity
- The most comprehensive temperature and heat flow calibration on the market
- Ease of use
- Unsurpassed temperature control using a 22 bit, ultra high resolution A/D converter (U.S. Patent 4734678).

The general approach utilized to obtain OIT values is to heat the sample under nitrogen from room temperature to an elevated temperature which is above the melting point of the polyethylene. The most commonly used temperature is 200°C and the sample is maintained at this temperature under an oxygen purge until the onset of degradation is observed.

When measuring the oxidative stabilities of polyethylenes, the following guidelines will help to provide the best possible results in terms of reproducibility:

Thermal Analysis & Surface Solutions GmbH
Pfingstweide 21
61169 Friedberg - Germany

+49-6031-16223-1
+49-6031-16223-29
Info@thass.net
http://thass.net

- Pre-check the DSC cell to ensure that the DSC has been accurately calibrated for temperature using high purity indium (M.P. = 156.6°C) and tin (M.P. = 232.0°C) standards.
- Pre-check the DSC cell to ensure that it will achieve an isothermal hold temperature of 200.0°C. (The temperature learn mode will help to accomplish this.) Deviations from the isothermal target temperature will significantly affect the measured OIT values.
- Pre-check the nitrogen and oxygen purge gases to ensure that a stable flow of the purge gases is achieved. The recommended purge gas flow rate is 100 mL/min when performing OIT testing.
- Use a sample mass of approximately 10 to 15 mg.
- Use open aluminum sample pans. The use of copper pans should be avoided.
- Better sample uniformity can be obtained by extracting the sample from the coating material using a cork borer which has a diameter matching that of the DSC sample pan.
- Heat the sample under a nitrogen purge adjusted to a flow rate of 100 mL/min from room temperature to 200.0°C at a rate of 10°C/min.
- Hold the sample under the nitrogen purge at 200.0°C for a 5 minute period to allow the sample and DSC cell to thermally equilibrate at the isothermal target temperature.
- During the initial heating and the 5 minute isothermal purge, data storage is recommended to be turned OFF. This provides a constant definition of time zero at the exact point at which the purge is switched from nitrogen to oxygen.
- Switch from the nitrogen purge to an oxygen purge after the 5 minute holding period using the Gas Switching Accessory. The flow rate of the oxygen purge should be a consistent 100 mL/min. Data storage should be ON.
- The sample should be maintained under isothermal conditions until a significant oxidative exothermic response is obtained. This may take up to 3 hours, depending on the relative stability of the polyolefin being tested. Once a significant exothermic onset has been obtained, the experiment can be terminated.
- Plot the resulting heat flow data as a function of time.
- The OIT value is defined as the onset time established by using the onset option contained in the DSC Analysis software. The initial point is placed in the flat portion of the DSC heat flow signal prior to the onset of oxidation. The end point should be placed in the sloping portion of the exothermic response.

Thermal Analysis & Surface Solutions GmbH
Pfingstweide 21
61169 Friedberg - Germany

+49-6031-16223-1
+49-6031-16223-29
Info@thass.net
http://thass.net

SII NanoTechnology Inc.

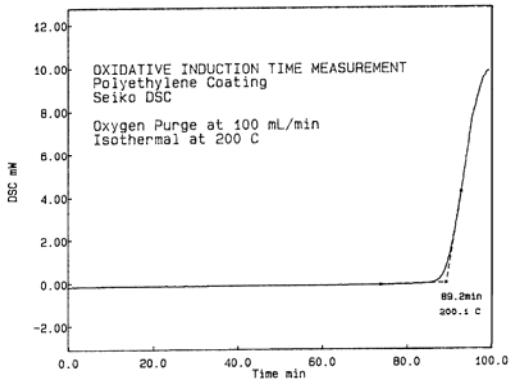

Figure 1

Figure 1 shows the DSC OIT results obtained on a polyethylene coating. The sample undergoes oxidative degradation at 200°C at 89.2 minutes, based on the measured onset time.

Thermal Analysis & Surface Solutions GmbH
Pfingstweide 21
61169 Friedberg - Germany

+49-6031-16223-1
+49-6031-16223-29
Info@thass.net
http://thass.net