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Application News

Differential Scanning Calorimeter DSC-60 Plus

Measurement of Oxidation Induction Time(OIT) of Deteriorated Polypropylene with a Differential Scanning Calorimeter

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User Benefits

- By measuring the oxidation induction time using a DSC, the degree of deterioration and the effects of antioxidants in rubbers, plastics, and other materials can be evaluated.
- By using "OIT Automatic Termination Software," measurements can be stopped and oxidation induction time analyzed automatically.

■ Introduction

Polymer materials, such as rubbers and plastics, absorb oxygen in the air during molding and use. As the oxidation reaction proceeds, it can cause various deterioration phenomena. Therefore, antioxidants or other additives are generally added to polymer materials to prevent oxidative deterioration. Evaluation of the effectiveness of antioxidants, resistance to heat and light, and the degree of deterioration is extremely important at the development and manufacturing stages. The effect of additives and the degree of deterioration can be evaluated by measuring the oxidation induction time (OIT) with a differential scanning calorimeter DSC. This paper describes the results of measuring oxidation induction time in polypropylene that has been deteriorated by heat treatment for different periods or deteriorated by irradiation with ultraviolet rays.

■ Oxidation Induction Time

Oxidation induction time (OIT) is the time during which heat generation occurs due to an oxidation reaction. To measure OIT, the temperature is raised to a target temperature in an inert atmosphere, the temperature is maintained, and then the inert atmospheric gas is replaced with oxygen or air. The oxidation induction time is defined as the period from when the atmosphere is replaced until the rise of the exothermic peak caused by oxygen absorption. As an example, Fig. 1 shows the results of measuring the oxidation induction time of polypropylene.

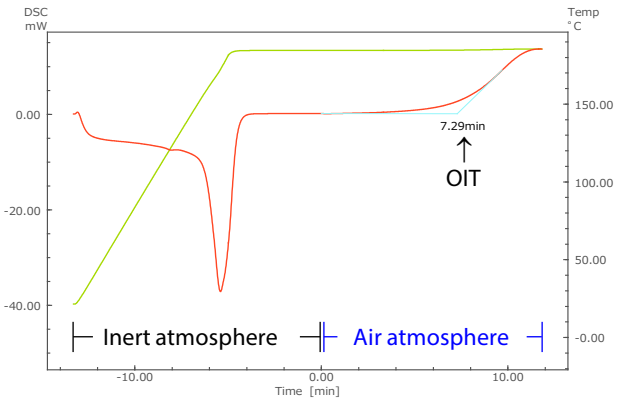

Fig. 1 Results of Measuring the Oxidation Induction Time of Polypropylene

In this measurement, it can be seen that oxidation occurs 7.29 minutes after raising the temperature to 185 °C in a nitrogen atmosphere, maintaining that temperature, and then switching to an air atmosphere.

By using the LabSolutions TA application program "OIT automatic termination software," it is possible to detect the peak due to oxidation, automatically terminate the measurement, and automatically analyze the oxidation induction time based on when the peak begins to rise.

■ Sampling and Measurement Conditions

To measure heat-treated polypropylene, a total of five samples were used, including four polypropylene samples that had been heat-treated at 150 °C for 456 hours, 624 hours, 744 hours, or 1080 hours, and one polypropylene sample that had not been heat-treated. To measure polypropylene irradiated with ultraviolet rays, a total of two samples were used, including a polypropylene sample irradiated with ultraviolet rays for 5 hours and a polypropylene sample not irradiated with ultraviolet rays. Fig. 2 shows the appearance of the polypropylene sample that was not heat-treated.

Fig. 2 Sample Appearance

Fig. 3 State of Sampling

Table 1 Measurement Conditions

Device:	DSC-60 Plus
Heating Rate:	20 °C/min
Temp. Program:	Increase temperature from room temperature to 215 °C and maintain
Sample Amount:	10 to 20 mg
Atmospheres:	N_2 50 mL/min before switching Air 50 mL/min after switching

Fig. 4 DSC-60 Plus Differential Scanning Calorimeter

■ Measurement Results from Heat-Treated Polypropylene

Fig. 5 and Fig. 6 show the measurement results of unheat-treated polypropylene and polypropylene heat-treated for 456 hours. In addition, Fig. 7 shows a graph of the relationship between the heat treatment time and oxidation induction time for each sample.

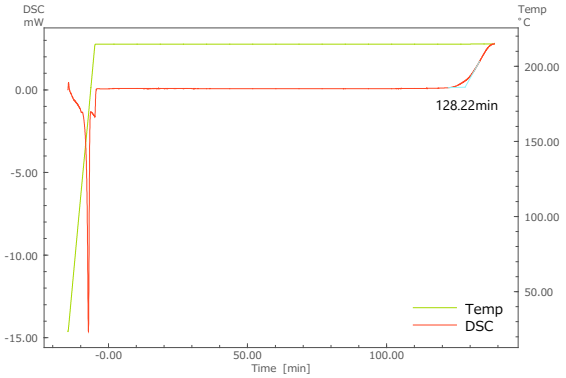

Fig. 5 Measurement Results of Unheat-Treated Polypropylene

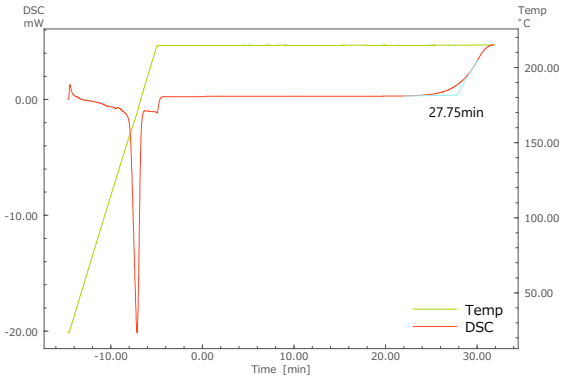

Fig. 6 Measurement Results of Polypropylene Heat-Treated for 456 Hours

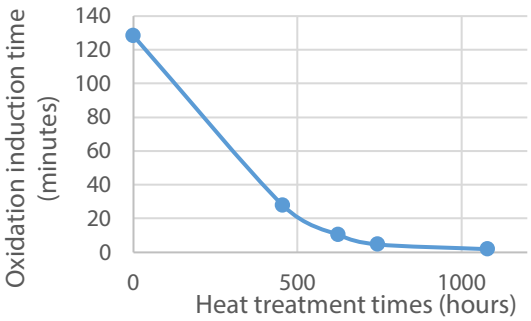

Fig. 7 Relationship between Heat-Treatment Time and Oxidation Induction Time for Each Sample

The results in Fig. 7 show that oxidation induction time decreased as sample heat treatment time was increased, indicating that heat treatment promoted deterioration of the sample and made it easier to oxidize.

■ Measurement Results from Polypropylene Irradiated with Ultraviolet Rays

Fig. 8 shows the measurement results of polypropylene not irradiated with ultraviolet rays and Fig. 9 the measurement results of polypropylene irradiated with ultraviolet rays for 5 hours.

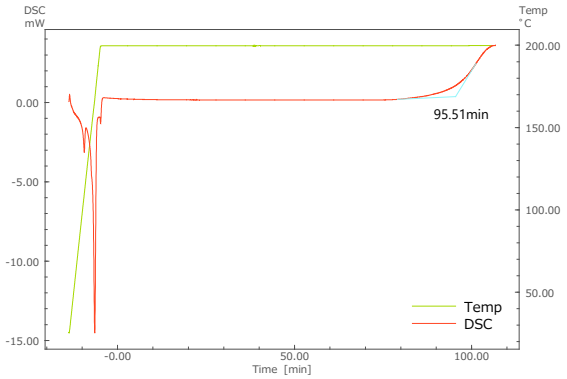

Fig. 8 Measurement Results of Polypropylene not Irradiated with Ultraviolet Rays

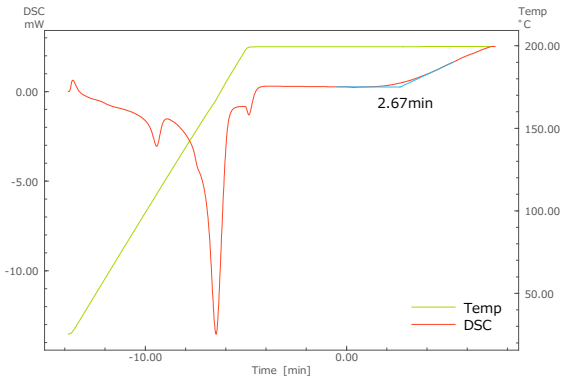

Fig. 9 Measurement Results of Polypropylene Irradiated with Ultraviolet Rays for 5 Hours

The results in Fig. 8 and Fig. 9 show an oxidation induction time of about 95 minutes for polypropylene not irradiated with ultraviolet rays, but an oxidation induction time of 2.67 minutes for polypropylene irradiated with ultraviolet rays for 5 hours, with oxidation starting immediately after replacing the nitrogen atmosphere with air. The above results indicate that 5 hours of UV irradiation sharply increased the deterioration rate.

■ Conclusion

By measuring the oxidation induction time of polypropylene with different heat treatment times and polypropylene irradiated with ultraviolet rays, it was possible to evaluate the degree of deterioration of the sample. As described in this article, DSC can be used to check the degree of deterioration of polymer materials and the effects of antioxidants by measuring the oxidation induction time.

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