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Application News

Differential Scanning Calorimeter
Simultaneous TG/DTA

Thermal Characterization of Virgin and Recycled PET

Atsuko Naganishi

User Benefits

- The DSC-60 Plus differential scanning calorimeter can evaluate the melting temperature and crystallization temperature of materials.
- Thermal stability can be evaluated by the DTG-60 simultaneous TG/DTA.
- Grasping the thermal properties of materials makes it possible to consider the optimum molding conditions.

■ Introduction

Recycled PET (polyethylene terephthalate) is an important material for a sustainable future. The use of renewable resources will be necessary to achieve the Sustainable Development Goals (SDGs). Recycled PET has a low environmental impact and contributes to the reduction of plastic waste. In particular, use of recycled PET will be an important step toward solving the problem of marine plastic pollution. Production of virgin PET requires fossil fuel, but consumption of fossil fuels can be reduced by using recycled PET, contributing to the reduction of greenhouse gas (GHG) emissions. Thus, recycled PET can contribute to the realization of a circular economy that reuses waste as a resource and the construction of a sustainable society.

In this paper, the thermal properties of virgin PET fiber and recycled PET fiber were evaluated using the DSC-60 Plus differential scanning calorimeter and the DTG-60 simultaneous TG/DTA.

Fig. 1 Appearance of DTG-60 (Left), and DSC-60 Plus (Right)

■ Samples

Fig. 2 Samples of Virgin PET Fibers (Left) and Recycled PET Fibers (Right)

■ Evaluation of Melting Behavior by DSC

To investigate the melting behavior of virgin and recycled PET fibers, the DSC-60 Plus (Fig. 1, right) and TAC-60i electric cooling device were used. The samples were heated from 0 °C to 300 °C at a rate of 10 °C/min (first heating), cooled to 0 °C at -10 °C/min (first cooling), and then heated again to 300 °C (second heating). Table 1 shows the analysis conditions.

Table 1 Analysis Conditions for Virgin and Recycled PET Fibers

Instruments	: DSC-60 Plus + TAC-60i
Heating and cooling rate	: 10 °C/min
Temperature range	: 0 °C – 300 °C
Sample weight	: 4 mg
Atmosphere	: Nitrogen

Recycled PET fibers can be produced by two methods, material recycling and chemical recycling. The recycled PET fibers measured in this study were produced by the former method. Fig. 3 and Fig. 4 show the heating and cooling data of the virgin and recycled PET fibers, respectively. Table 2 summarizes the peak top temperatures of first heating, first cooling, and second heating. The melting peak shape and melting temperature of the virgin and recycled PET fibers were different at first heating. The crystallization temperature at cooling and the melting temperature at second heating were also different, confirming that the thermal behavior of virgin and recycled PET fibers is different. PET is normally a linear polymer obtained by condensation polymerization of ethylene glycol and terephthalic acid. In PET bottles, part of terephthalic acid is replaced with isophthalic acid to control crystallization. Since the recycled PET fibers in this study were derived from PET bottles, the decrease in the melting temperature at first heating is considered to be due to the isophthalic acid. The two endothermic peaks at second heating may be due to the thermal history or recrystallization during heating. It is important to understand the thermal properties during molding.

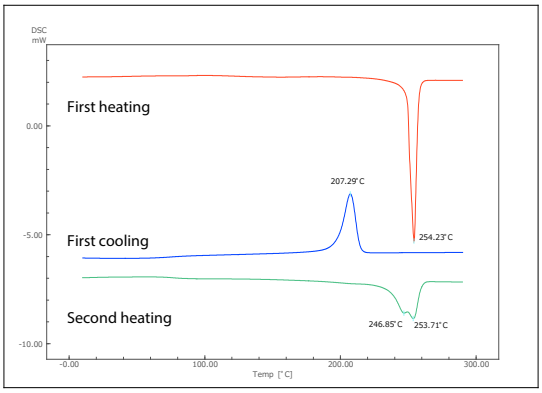

Fig. 3 DSC Curve of Virgin PET Fiber

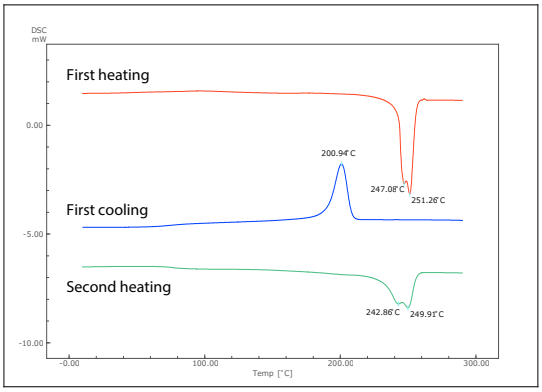

Fig. 4 DSC Curve of Recycled PET Fiber

Table 2 Peak Temperatures of Virgin and Recycled PET Fibers

Sample	First heating (melting temperature)	First cooling (crystallization temperature)	Second heating (melting temperature)
Virgin PET fiber	254.2 °C	207.3 °C	246.9 °C 253.7 °C
Recycled PET fiber	247.1 °C 251.3 °C	200.9 °C	242.9 °C 249.9 °C

Next, heating and cooling of virgin PET fibers were repeated six times. Table 3 shows the analysis conditions. Fig. 5 shows the DSC curves from first heating to sixth heating. Since first heating was affected by the thermal history, the low-temperature endothermic peak temperatures of the two endothermic peaks observed from second heating to sixth heating were compared. Fig. 6 shows the results. As the number of heating cycles increased, a linear decrease in the melting temperature was observed, indicating a reduction in crystallinity. This suggests that PET fibers may undergo changes in physical properties with repeated heating.

Table 3 Analysis Conditions for Repeated Heating and Cooling of Virgin PET Fibers

Instruments	: DSC-60 Plus + TAC-60i
Heating and cooling rate	: 10 °C/min
Temperature range	: 0 °C - 300 °C
Sample weight	: 4 mg
Atmosphere	: Nitrogen

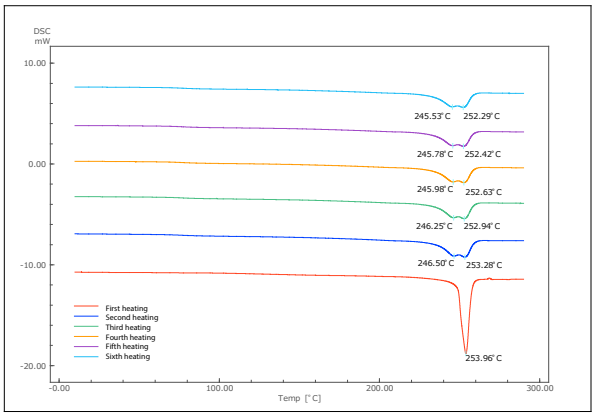

Fig. 5 DSC Curve of Repeated Heating and Cooling of Virgin PET Fibers

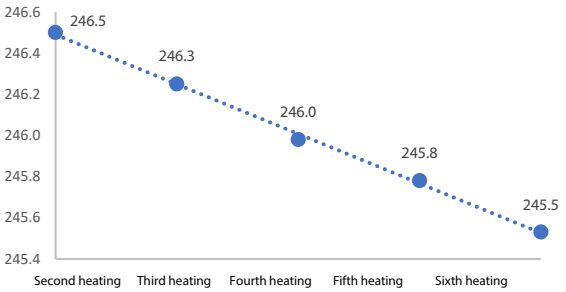

Fig. 6 Low-Temperature Melting Point in Repeated Heating and Cooling of Virgin PET Fibers

■ Evaluation of Decomposition Behavior by TG-DTA

Finally, virgin PET fiber and recycled PET fiber were heated in nitrogen (Fig. 7) or air (Fig. 8) using a DTG-60.

Table 4 shows the analysis conditions. In nitrogen, both fibers began to show weight loss at around 400 °C, and no differences in heat resistance were observed. However, a difference of about 5.7 % was confirmed in the weight loss rate from 30 °C to 550 °C, indicating differences in residues. In air, no difference in residues was observed, but the behavior of the second stage of weight loss was different.

Table 4 Analysis Conditions for TG-DTA Measurements of Virgin and Recycled PET Fibers

Instruments	: DTG-60
Heating and cooling rate	: 20°C/min
Temperature range	: 30°C - 600°C
Sample weight	: 4 mg
Atmosphere	: Nitrogen or Air

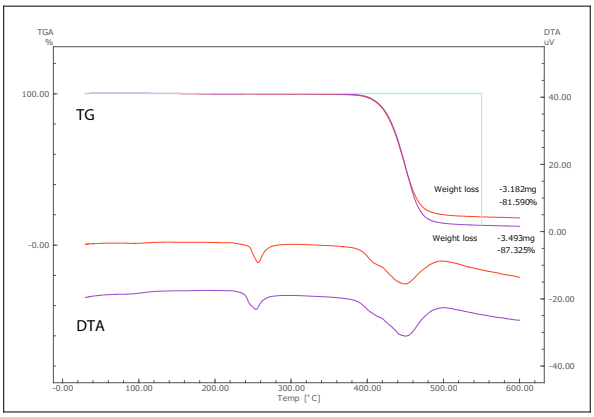

Fig. 7 TG-DTA Curves of Virgin and Recycled PET Fibers in Nitrogen

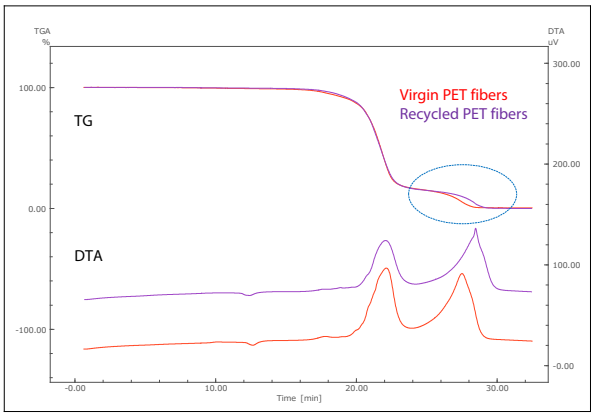

Fig. 8 TG-DTA Curves of Virgin and Recycled PET Fibers in Air

■ Conclusion

The thermal properties of PET fibers were evaluated using the DSC-60 Plus differential scanning calorimeter and the DTG-60 simultaneous TG-DTA. Differences in the melting temperatures of the virgin and recycled PET fibers were evident, and it was shown that repeated heating leads to a decrease in crystallinity. While both fibers exhibited the same heat resistance, differences in residues were observed when the samples were heated to 600 °C. Understanding these thermal characteristics enables optimization of fiber processing and improvement of product quality. Additionally, knowledge of the thermal properties of recycled PET fibers will make it possible to promote the use of sustainable materials, facilitating the development of products that minimize environmental impacts.

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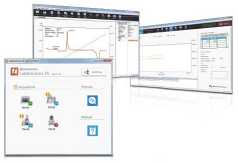

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