Evaluation for Melting Behaviour of Polymer Materials using Sample Observation DSC System

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Introduction

Since a sample is put in the furnace generally, it's too difficult to directly observe the sample behavior visually during measurement. Therefore, we have suggested a thermal analyzer equipped with camera function to easily understand the sample's reaction recently[1].

Same polymer materials with different molecular weight were measured. Shapes of the melting peak were different between low molecular and high molecular. The molecular weight become smaller, the melting temperature shows lower in general.

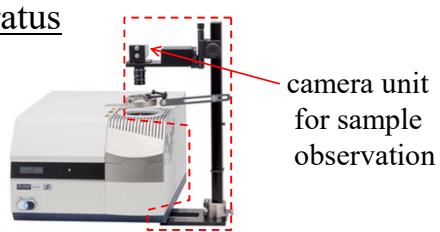
In this study, For the quantitative discussion of the images observed by DSC measurement, we evaluated the brightness and the surface be from these images.

Samples and Measurement method

Samples

- **Polypropylene**(Pellet)
  Mw 12000
  Mw 250000
  Mw 340000
- **Polyethylene**(Pellet)
  density 0.945
  density 0.958

Apparatus

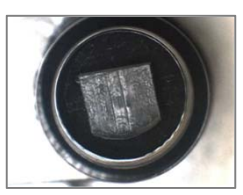


DSC7000X High Sensitivity Differential Scanning Calorimeter
RV-1DX Real View® Sample Observation Unit

Measurement method

Measurement Conditions
- Temperature range : R.T. to 200 ºC
- Heating rate : 10 ºC/min
- Sample weight : 5 mg
- Sample pan : Sapphire open pan

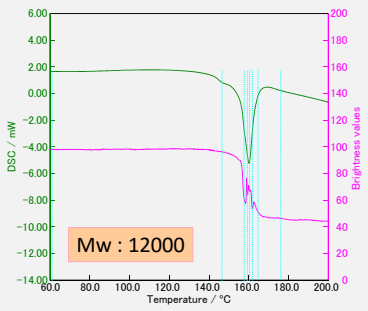
Sapphire pan restrains the luster and the reflection of pan.

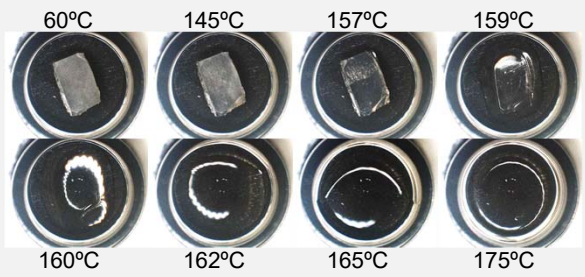


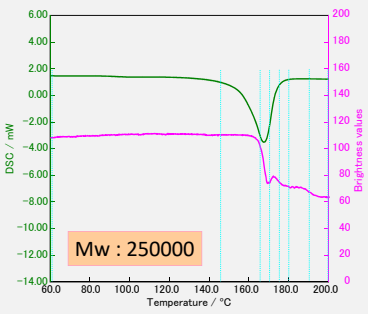
Sample and Sapphire pan

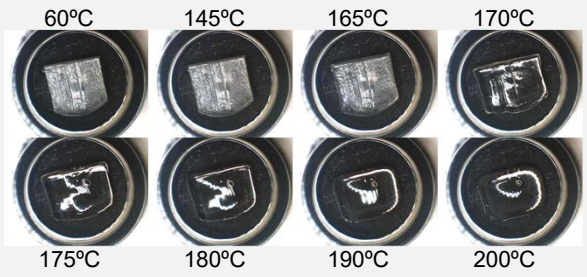
Results

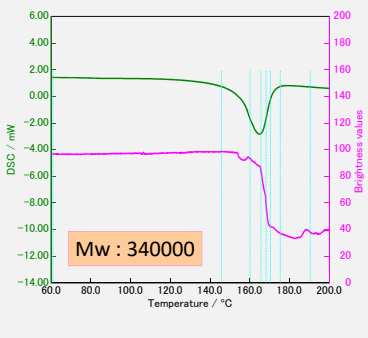
Polypropylene

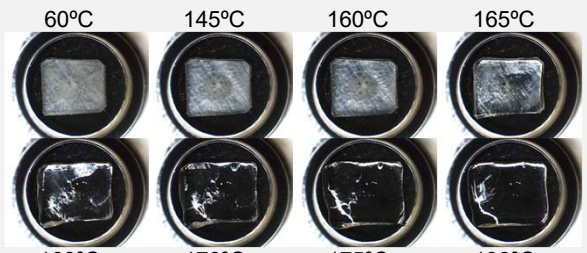




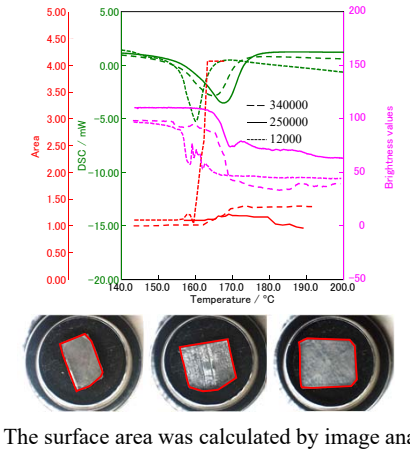








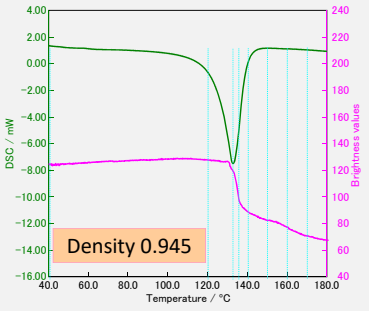
Mw12000 has decreased brightness on the low temperature side of the melting peak. The change of the sample shape is dynamic, and disorder is observed for a change of the brightness, on the images around peak-top temperature.
The change of the sample shape of Mw250000 and 340000 begins over the peak-top temperature. Afterwards, difference is observed for the flow behavior of the sample, and the brightness shows a graded change.
The change of the brightness shows transparency (colorless) accompanied with the melting. Also it is considered that luster of the sample surface is appeared with change of the sample shape.



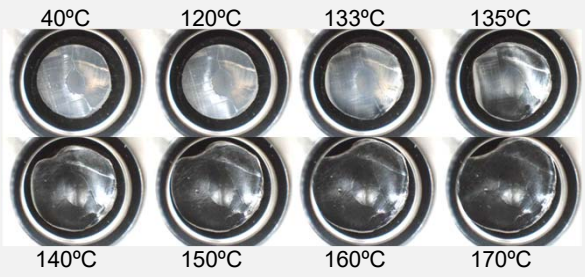
The surface area was calculated by image analysis software, as the initial surface area of specimen is one.

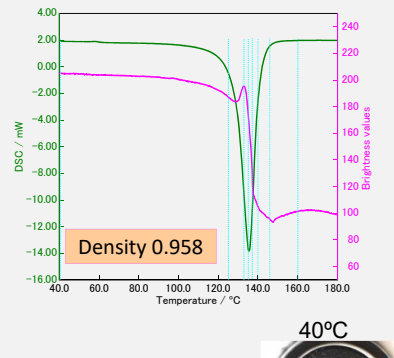
Evaluation of the surface area of the specimen was tried to perform quantification of a difference of the inconstancy of the shape in Polypropylene.
The surface area of Mw12000 increased near the peak top temperature. because of the specimen flows and spread.
The surface area of Mw250000 slightly increase early in a peak temperature. However, after the peak, surface area decreased by change into a spherical shape.
Because it spread with keeping the form of the board, the surface area of Mw340000 increased after the peak.

Polyethylene

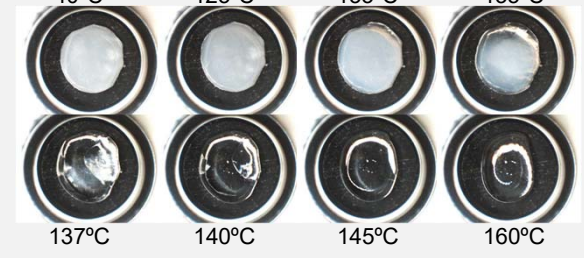


From an observation image of the sample density with 0.945, the specimen flows a little and spreads after the melting.
The decrease of the brightness confirm two phases and may show the graded flow behavior.





As for 0.958 of the density, a decrease of the brightness is observed on the low temperature side of the melting peak, in comparison with the density of 0.945. At the high temperature side of the peak, brightness increase slightly. It is considered that the change to spherical shape of the sample depends on the specimen flowing with melting.



Summary and Conclusion

- A quantitative evaluation of the color change (colorless) accompanied with the melting is possible by calculating the brightness from the observation image of the specimen.
- A shape change and the flowing behavior accompanied with the melting of the specimen are estimated by the evaluation of the change of the brightness. However, the influence of the reflection of the light by becoming surface luster must be considered .
- The lower molecular weight and the lower density tended to be easy to flow by melting.

Reference

[1] E. Shimoda, Y. Kasai, M. Iwasa, Y. Nishiyama, The 51st Japanese Conference on Calorimetry and Thermal Analysis, The Japan Society of Calorimetry and Thermal Analysis, (2015) 139