

Crystallization Measurement by Optical Observation DSC using a Polarizing Filter

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Introduction

Hitachi thermal analysis systems can observe the sample during measurement and pinpoint when visible changes occur. It is possible to simultaneously obtain optical images and collect thermal data, such as DSC and TG, and to link the data together. As a result, we can more thoroughly understand the phenomena during thermal analysis.

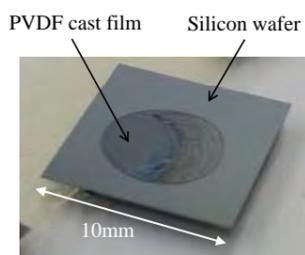
To enhance the sample observation information collected using a DSC, a polarizing filter was installed, and images of the spherocrystals in polyvinylidene fluoride (PVDF) were successfully collected.

The configuration and the results from this new optical observation system design are presented, along with discussion of the measurement results.

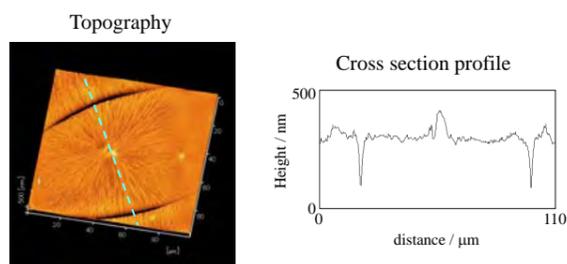


Differential scanning calorimeter DSC7000X with Real View System

Sample

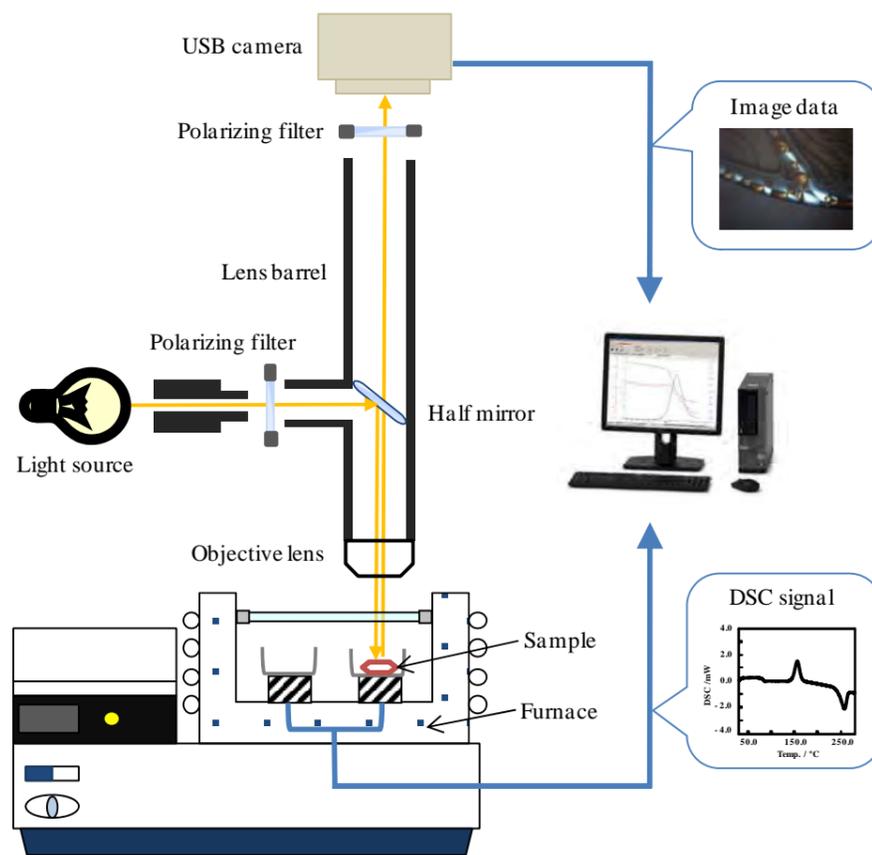


Surface observation of PVDF cast film by SPM



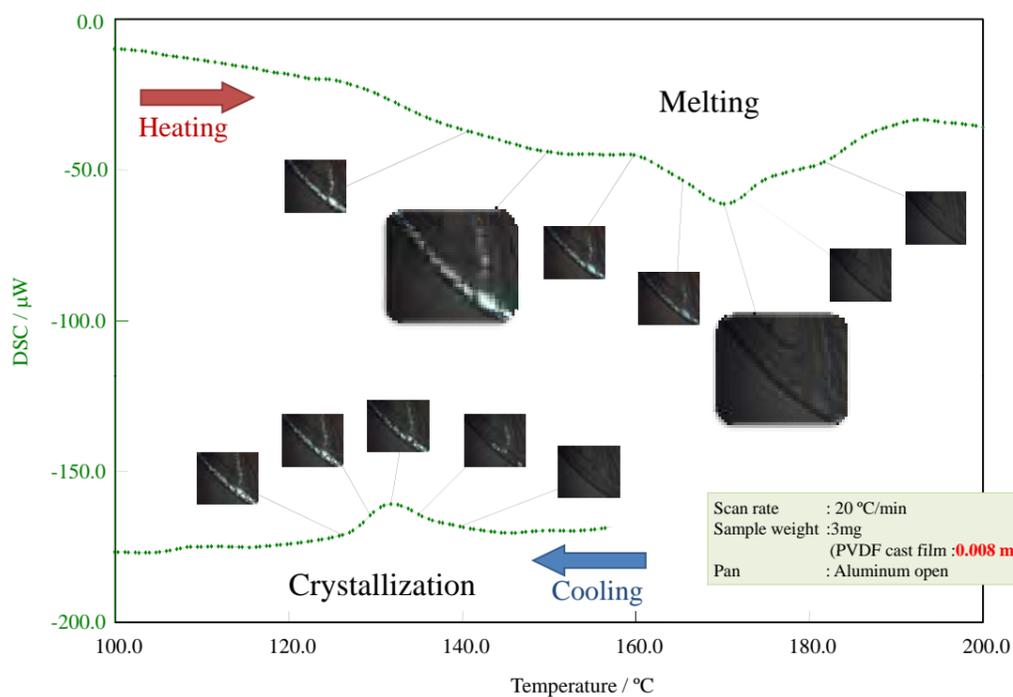
- Melting of PVDF is a reversible phenomenon. It can be done repeatedly and reversibly.
- Sample was cut to about 3mm in order to fit into the sample pan

Configuration



Results

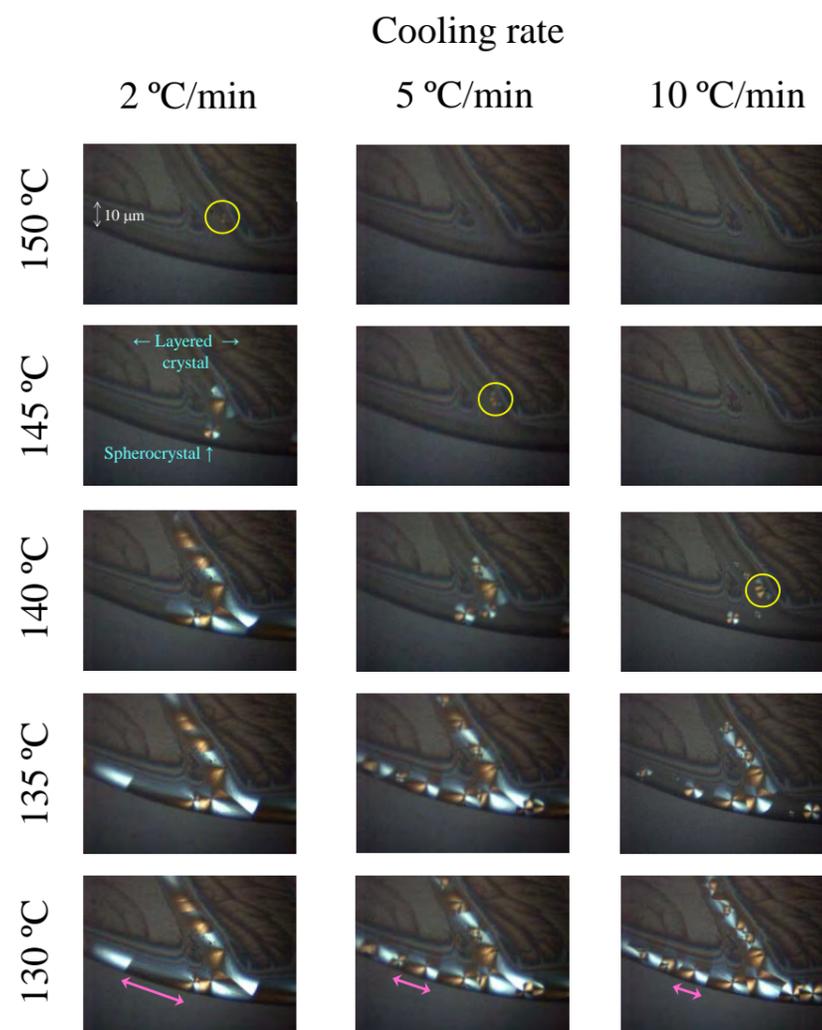
★ An example of optical observation DSC data using a polarizing filter



Each point on the DSC curve represents where one image was collected. Most of the 3mg sample weight was silicon wafer. However the melting and crystallization peak of PVDF were clearly detected even though its weight contribution was extremely small.

These image data show the crystallization process of melted PVDF. It is found that the spherocrystal size is affected by the cooling rate. The spherocrystal becomes large as cooling rate becomes low. However the physical location of crystallization is the same.

★ Cooling rate dependence of crystal size



Conclusions

- Hitachi has demonstrated thermal analysis combined with sample observation using a polarizing filter. Furthermore, this DSC system has the capability of detecting a peak even if sample weight is extremely small.
- The measurement results show that the size of spherocrystal depends on the cooling rate after melting.

Acknowledgment

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