# Isothermal Crystallization by Rapid Heat-Cool DSC

## Introduction

As a technique, interest has been growing in performing differential scanning calorimetry (DSC) at higher than typical (10 °C/min) temperature-scanning rates. This is because a variety of material characterization challenges exist that can benefit dramatically from rapid heating or cooling rate experiments. For example, the investigation of metastable states and time-dependent transitions would profit greatly from fast scanning rates. In general, higher scanning rates will also increase the heat flow sensitivity for subtle transitions although this benefit is usually tempered by the small mass requirement of the rapid scanning rates.

A DSC has been designed specifically for operation at high scanning rates – up to 2000 °C/min in heating with similarly high cooling rates.[1] Key technologies introduced by TA Instruments are essential to, and have been incorporated into the instrument known as Project RHC. For example, Tzero technology improves the resolution and the sensitivity of the measured sample heat flow rates, especially for very weak effects, and improves the instrument baseline. Also, infrared heating, introduced in the Q5000IR TGA, provides a "massless" infrared heat source. Readers interested in further details on the instrument design should refer to reference 1.

This applications note reports on the study of polyethylene by isothermal crystallization.

## Results and Discussion

Isothermal crystallization is a time-to-event experiment. A typical method consists of raising the sample to be studied above its melt temperature and holding isothermal for a couple of minutes to ensure that the sample is completely melted. The sample is then rapidly cooled and stabilized at the desired test temperature. It is critical to not only bring the sample to the test temperature quickly, but also to not undershoot the test temperature. The Project RHC DSC is ideal for this type of experiment. The cooler design consists of a bath of liquid nitrogen in contact with the sample cell for ballistic cooling rates in excess of 2000 °C/min. The IR furnace design ensures that the temperature control is very fast and nimble when equilibrating the sample at the test temperature.

Polyethylene is a very fast crystallizing polymer and therefore a challenging material to analyze by isothermal crystallization. However, the Project RHC DSC is able to generate excellent data as illustrated in figure 1. The figure shows that at the lowest test temperature of 105 °C, the system stabilizes in less than 0.25 minutes (15 seconds) and fully captures the crystallization exotherm, which peaks at 0.3 minutes (18 seconds). The system also shows excellent reproducibility as the plot displays two runs at a test temperature of 110 °C.

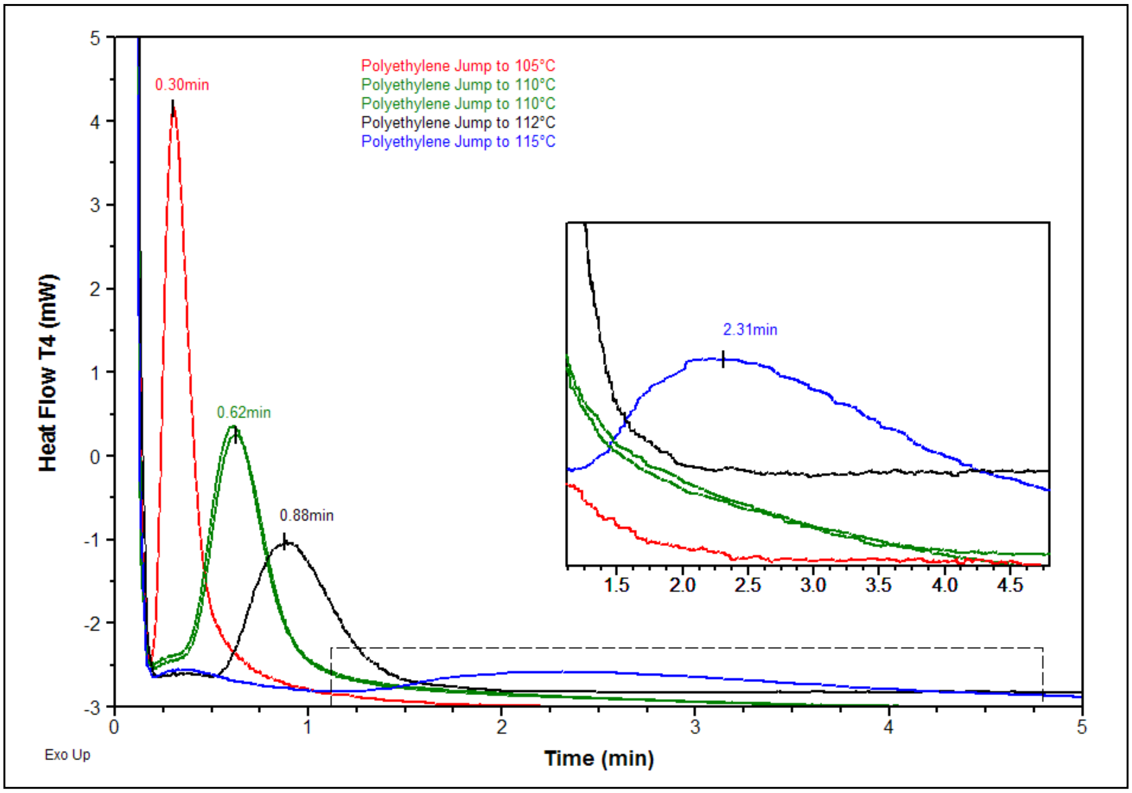


Figure 1 – Isothermal crystallization plots at 105, 110, 112 and 115°C.

## REFERENCES


1. Robert L. Danley, Peter A. Caulfield and Steven R. Aubuchon, *American Laboratory*, January 2008, pp. 9-11.


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