



DUBBLE — EXPERIMENT REPORT

Beam time number: 26-02 690		File number: 34223
Beamline: BM26-B	Date(s) of experiment: 22/04/2014 – 24/04/2014	Date of report: 17/09/2014
Shifts: 6	Local contact(s): G. Portale	

1. Who took part in the experiments?

Martin van Drongelen¹

Harm Caelers¹

Affiliation: 1. Material Technology Group, Department of Mechanical Engineering, Eindhoven University of Technology, the Netherlands.

Were you able to execute the planned experiments?

YES. All the planned experiments were performed. We were able to monitor the structure evolution during melt crystallization in multiple isothermal and non-isothermal conditions for isotactic polypropylene (iPP) and linear low-density polyethylene (LLDPE) resins.

2. Did you encounter experimental problems?

YES. We encountered temperature control problems of the sample due to problems with the nitrogen dewar. Fortunately, this could be fixed by the users after a certain time. In addition, we encountered some problems with simultaneous acquisition of SAXS and WAXS patterns due to incompatibility of the macro with the recently updated ESRF server software. In principle, series were programmed with short and long acquisition time, respectively. As a result of these unexpected problems, 80% of the

long acquisition time patterns was obtained with the *short* acquisition time, inducing a significant loss of frame quality. After data analyses, we managed to obtain all results desired, although this is not without data scatter.

3. Was the local support adequate?

YES. The support of the local contact, G. Portale and the technical staff, was needed to accurately set up the experimental equipment, see Figure 1. Moreover, their support was needed to implement the software needed for high-speed and simultaneous acquisition of SAXS/WAXD patterns.

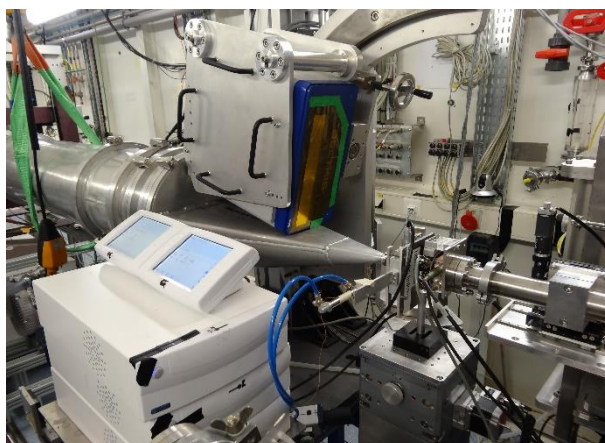


Figure 1: Temperature jump-stage installed in the experimental hutch of DUBBLE.

4. Are the obtained results at this stage in line with the expected results as mentioned in the project proposal?

YES. We were able to rapidly transfer the sample to a certain temperature below melting temperature and study the occurrence of crystal growth (WAXS) and formation/growth of lamellar entities (SAXS) during high cooling rates or controlled isotherms.

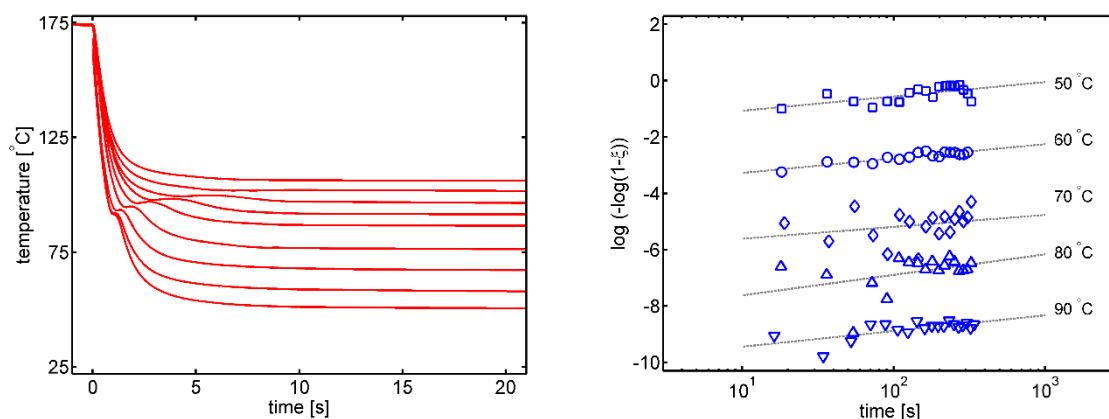


Figure 2: (a) LLDPE sample temperature as recorded by the embedded thermocouple and (b) Avrami plot of the obtained crystallinity indicative for the growth dimensionality during secondary isothermal crystallization.

The combination of our simple experimental setup with the high-frequency possibilities of the DUBBLE beam-line provided data on primary and secondary crystallization which has proven essential -in addition to the knowledge already gained from other methods- to fully describe structure formation kinetics of the examined LLDPE(s).

In case of the iPP the focus was mainly on the relation between crystallization temperature and the resulting lamellar thickness during non-isothermal crystallization. Due to the diversity in applied cooling rates this relation was found over a broad temperature range. Besides this relation, obtained from the SAXS measurements, the WAXS experiments were used for the investigation of the crystallization kinetics of a new iPP polymer grade. All data required to fit the crystallization model currently used in our group were obtained.

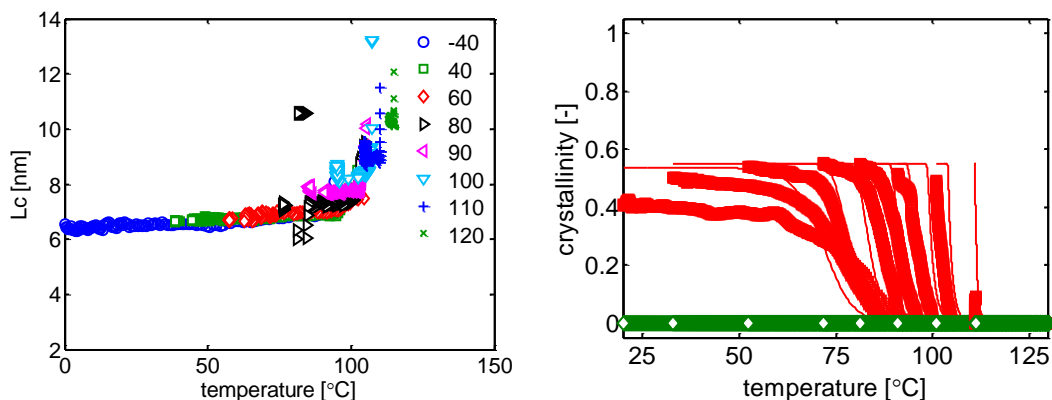


Figure 3: (a) Relation between crystallization temperature and resulting lamellar thickness as obtained from the SAXS measurements and (b) Crystallinity as a function of temperature for different cooling rates.

5. Are you planning follow-up experiments at DUBBLE for this project?

NO. At this point, none are scheduled and the current results are sufficient for implementation in our models.

6. Are you planning experiments at other synchrotrons in the near future?

NO.

7. Do you expect any scientific output from this experimental session (publication, patent ...)

YES. Two publications are currently in preparation including data of this experimental session. One manuscript is on the characterization of (melt-) crystallization kinetics of LLDPE. The second manuscript deals with describing mechanical properties as function of processing conditions.

8. Additional remarks

NO