DUBBLE – EXPERIMENT REPORT

Beam time number: 26-02/867		File number: 79806
Beamline: BM26-B	Date(s) of experiment: 29/05/2018 to 03/06/2018	Date of report: 09 May 2019
Shifts: 15	Local contact(s): Daniel Hermida Merino	

1. Who took part in the experiments?

Prakhyat Hejmady[1], Fabio Paolucci[1], Jessica pepe[1] , Coen Clarijs[1]

Affiliation:

1. Polymer Technology, Materials Technology Group, Department of Mechanical Engineering, Eindhoven University of Technology, the Netherlands.

Were you able to execute the planned experiments?

YES. We were able to perform all the planned experiments.

2. Did you encounter experimental problems?

NO. The setup and the beamline instrumentation were correctly working.

3. Was the local support adequate?

YES. The support of the local contact, D. Hermida Merino and of the technical staff, was adequate and allowed us to setup and run our experiments.

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

YES. All the experimental date collected at BM26-B will be used to understand the influence of processing conditions on crystallization kinetics during laser sintering. The outcome of the experiments is briefly described below.

Introduction

Our novel in-house developed experimental laser sintering setup, designed and constructed at the Polymer Technology section, TU Eindhoven, has the unique ability to allow in-situ time-resolved microscopic and X-ray observations of laser sintering with precise control over all sintering parameters. Since structure evolution and crystallization kinetics during laser sintering are complex and coupled processes involving non-isothermal, non-homogeneous temperature profiles and complex flow fields, this laser sintering setup provides direct access to essential local and time-resolved information about the structural processes involved. Experiments using this setup can provide us with essential knowledge to understand the relations between sintering conditions and microstructure development. The study on the sintering of PA12 particle pairs performed at ESRF demonstrates that real-time information about the sintering dynamics as well as the crystallization kinetics can be obtained with a good signal-to-noise ratio and with sufficient spatial and temporal resolution. Since the experiments performed at ESRF show the feasibility of the approach, we can conclude that our unique device opens up new promising perspectives in the field of 3D printing by selective laser sintering.

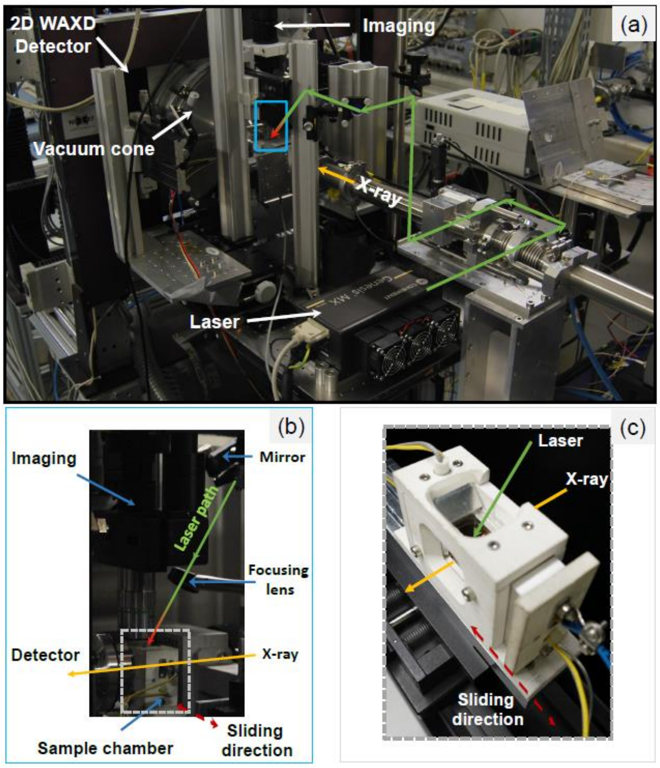


Figure 1 : Photograph of the experimental setup installed in the BM26 beamline at ESRF. (a) The setup is mounted on a x-y-z translator for positioning relative to the 2D WAXD detector and incident X-ray beam. (b) Inset picture shows the sample chamber with objective lens and vacuum cone. (c) Sample chamber.

Results

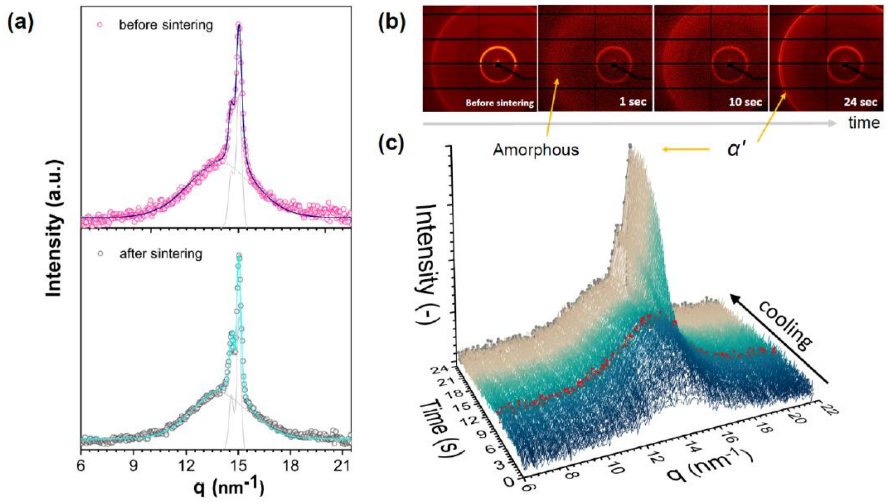
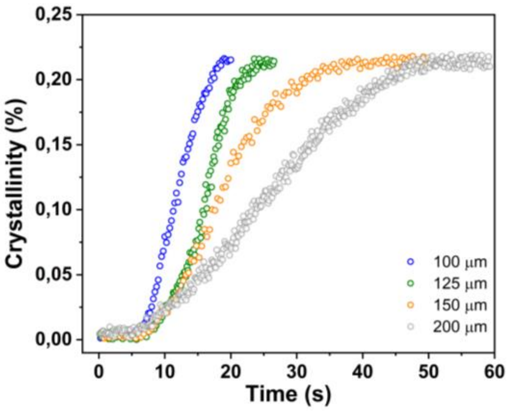


Figure 2 (a) integrated intensity as a function of scattering vector q, before and after sintering. The markers represent the radially integrated pattern obtained from experiments and the lines represent the sum of fitted peaks using a Gaussian-Lorentzian function. The grey dotted line is the amorphous halo and the grey solid line is the peak fit of the crystalline peak. (b) Image sequence of time-resolved 2D-WAXD patterns of PA12, before and during sintering for PA12 particles of radius a_o = 115 µm.(c) 3D plot of the radially integrated intensity as a function of scattering vector q for the complete acquisition time. Red line represents the amorphous halo before crystallization starts.

To demonstrate how laser sintering affects the crystal morphology, the intensity profile before sintering is compared with that after sintering (figure 2a). Figure 2(c) shows these intensity profiles, obtained from radially integrating the intensity over an azimuthal angle of 90° (figure 2b) and plotting it versus scattering vector q as a function of time up to the point at which steady state was reached. The integrated intensity, peak position, and peak width were obtained by fitting the curve with a double Gaussian-Lorentzian function. The crystallinity was calculated from the deconvolution of the total intensity into the amorphous and crystalline contributions.



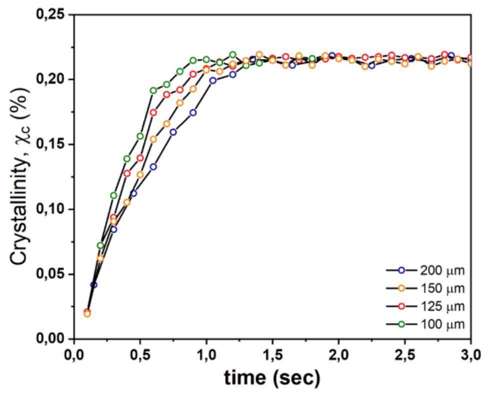


Figure 3 Influence of particle size on crystallization kinetics during sintering of PA12 particle doublets, chamber temperature (left) 155 °C (right) 110 °C

Similar experiments were performed for varying processing conditions like particle size and chamber temperature, as shown in figure 3. Clear differences can be observed in crystallization kinetics that can only be studied via such in-situ experiments. These experimental data from a case study on PA12 particles, which demonstrate the unique capability of our setup to capture the kinetics of the development of the macrostructure in the form of neck radius growth as well as the microstructure in the form of crystallinity and crystal-type evolution during laser sintering. The results also illustrate the accuracy with which these features can be obtained. Future experiments at different processing conditions for different classes of polymers ranging from commodity polymers like polystyrene to high performance polymers like polyether ether ketone can be sintered in the setup. The setup is also flexible towards the use of small angle X-ray scattering or more local characterizations using a more pronounced focusing of the X-ray beam.

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

Yes, we hope to perform more experiments once the beamline is open in 2020.

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 expected from this work. A scientific outcome (publication) describing the setup and proof-of-concept of the in-situ X-ray studies during laser sintering has already been submitted to the international peer-reviewed journal Review of Scientific Instruments. Another publication that focusses on the effects of processing conditions on the crystallisation process of Pa12 during laser sintering is in preparation.

8. Additional remarks

DUBBLE - CLAIM FORM FOR COSTS OF TRAVEL/SUBSISTENCE

Dutch users of beam time at DUBBLE can use this form to claim full/partial reimbursement of the associated costs of travel and subsistence. The form must be returned to NWO **within 2 months of the completion of the experiment** to **dubble@nwo.nl**

Reimbursement rules (costs are reimbursed to the Main Proposer)

Travel costs
€ 400 p.p. for max. 3 persons.

Subsistence costs
Subsistence costs are reimbursed for max. 3 persons @ € 60 p.p. per day (incl. 1 day before the experiment).

Applicant (Main Proposer) : Gerrit W.M. Peters

Beam time number : 26-02/867

Experiment dates : 29/05/2018 to 03/06/2018

Participants (max 3 persons):

Name : Prakhyat Hejmady
Name : Fabio Paolucci

Name : Jessica Pepe

Payment details

Pay to account no.: NL42RABO0158249658 (Project Nr. 353000/10018571)

Name: TECHNISCHE UNIVERSITEIT EINDHOVEN

City: Eindhoven