DUBBLE – EXPERIMENT REPORT

Beam time number: 26-02-893		File number: 82247
Beamline: BM26-B	Date(s) of experiment: 03/12/2018 to 07/12/2018	Date of report: 21 June 2019
Shifts: 9	Local contact(s): Daniel Hermida Merino	

1. Who took part in the experiments?

Prakhyat Hejmady[1], Jessica pepe[1] , Stan Looijmans [1]
Affiliation:

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

Were you able to execute the planned experiments?

No. We were not able to perform all the planned experiments due to maintenance of the X-ray beam. Due to unforeseen safety check at ESRF and regular loss of beam intensity delayed the work. Due to this we had only 18 hours to perform our measurements, in which 3 hours was used to disassemble the setup before the next user.

2. Did you encounter experimental problems?

The setup and the beamline instrumentation had no problems. Regular loss of beam intensity hindered our experiments.

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 run the experiments.

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

YES. The experimental data collected at BM26-B allowed us to understand the influence of processing conditions on the crystallization kinetics of polymer nanocomposites. Part of the outcome of the experiments is briefly described below.

Experimental

We studied the laser sintering of PA12 particle doublets using a novel in-house developed experimental setup, which mimics the main features of an SLS machine with the capability to perform in-situ X-ray experiments. By simultaneously capturing the WAXD patterns as well as optically following the dynamics of the bridge growth in between both particles, essential knowledge about the relations between sintering conditions and microstructure development was obtained. This data features information on morphological changes during the sintering process, which can only be studied by *in-situ* measurements. Experiments were conducted on PA12 and PA12 enriched with carbon nanotubes (CNTs) to understand the influence of additives on the crystallization with pure PA12 setting the benchmark.

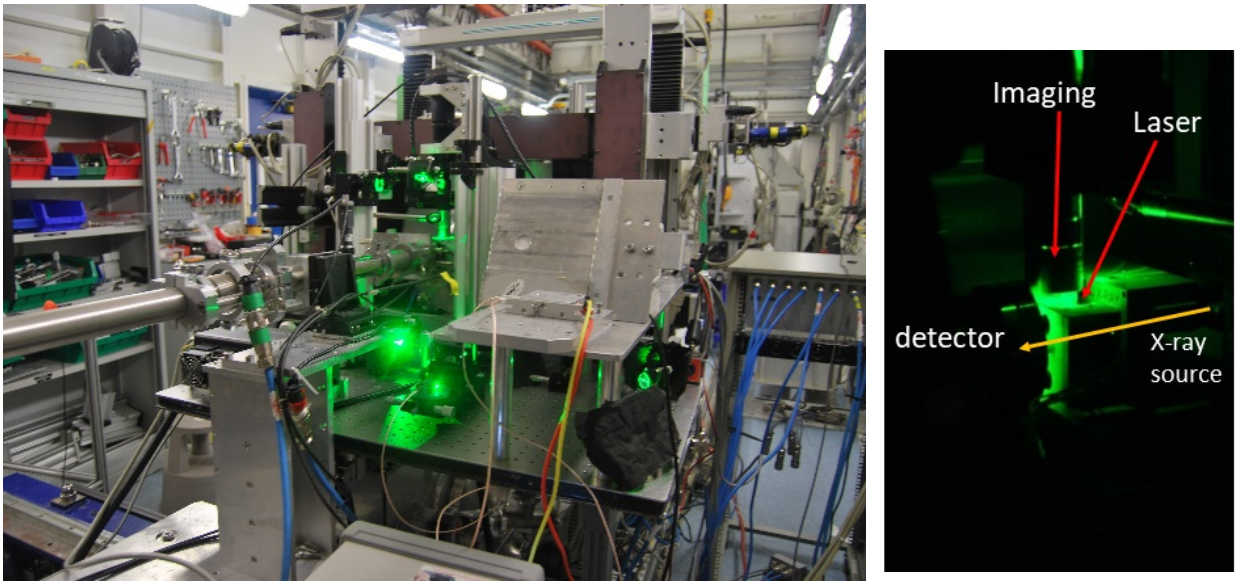

Figure 1 : (Left) Photograph of the experimental setup installed in the BM26 beamline at ESRF. (Right) In-situ experiments with laser and X-ray source to study sintering.

Results

To understand how filler content, in this case CNTs, affects the crystallization kinetics, laser sintering experiments on PA12 particles were performed for varying particle diameters and different wt% of CNTs. Figure 2 shows the intensity profiles, obtained from radially integrating the intensity over an azimuthal angle of 90^{o} and plotting it versus scattering vector q as a function of time. Time zero corresponds to the moment at which the laser was switched on and data was collected up to the point at which steady state was reached.

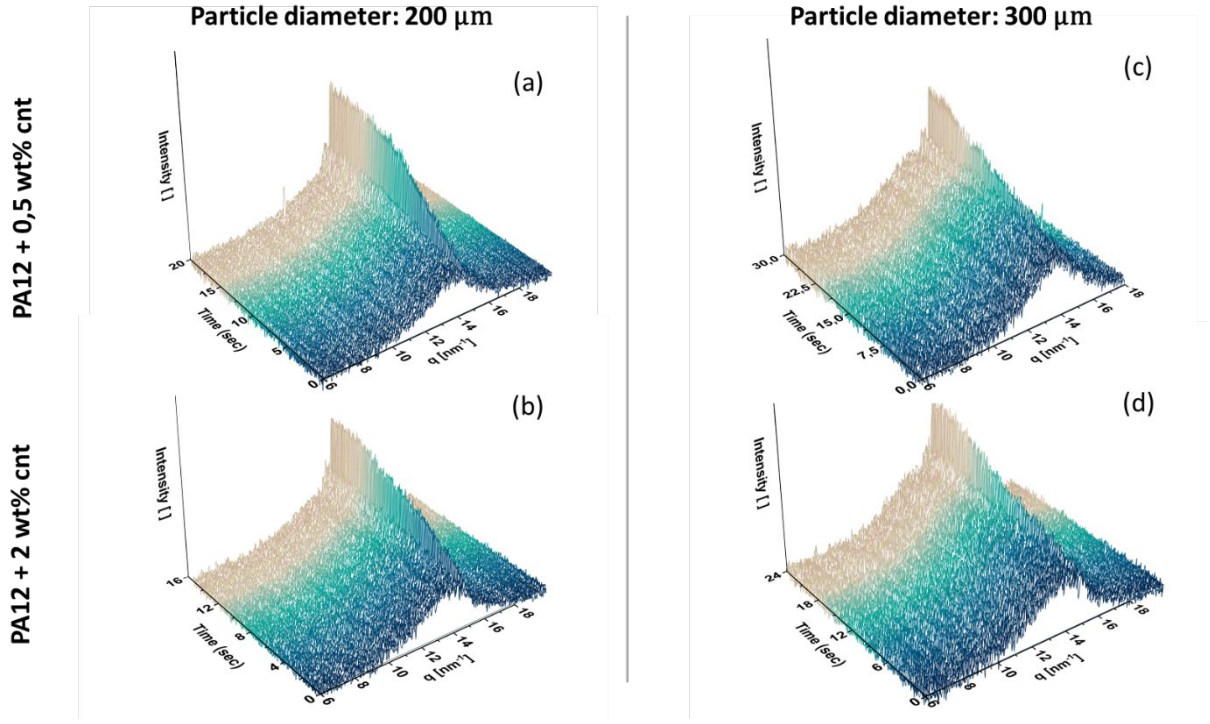

Figure 2 : 3D plot of the radially integrated intensity as a function of scattering vector q for the complete acquisition time. Figure 2(a) and (c) represent experiments performed for PA12 particles with 0.5 wt% CNTs and Figure 2(b) and (d) for 2 wt% CNT.

To evaluate the percentage crystallinity for these experiments, the integrated intensity, peak position, and peak width were obtained by fitting the curves with a double Gaussian-Lorentzian function. The crystallinity was calculated from the deconvolution of the total intensity into the amorphous and crystalline contributions as shown in figure 3. It can clearly be seen that the CNTs enhance the crystallisation kinetics. Also, a comparison was made by comparing WAXD pattern of before and after sintering, for the given processing condition. Figure 4 shows an example wherein it can be seen that the shoulder transforms into a peak at q= 14.5 nm^{-1}. This structure, also known as α'-phase, is the stable phase at high temperature for polyamide 12 polymer.

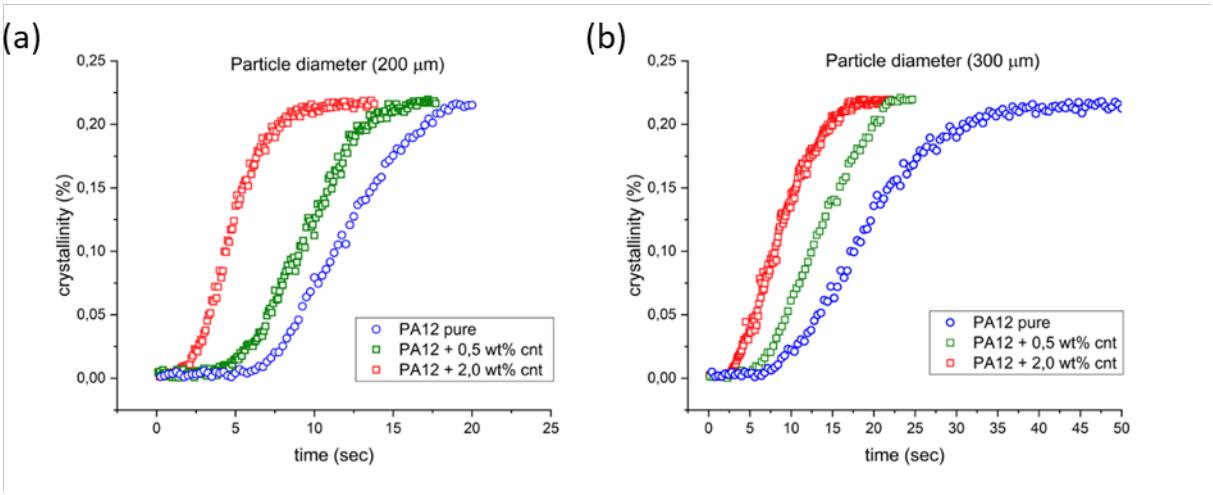

Figure 3: Influence of CNT content on crystallization kinetics during sintering of PA12 particle doublets at a chamber temperature of 155 °C . (a) particle diameter of 200 µm and (b) 300 µm.

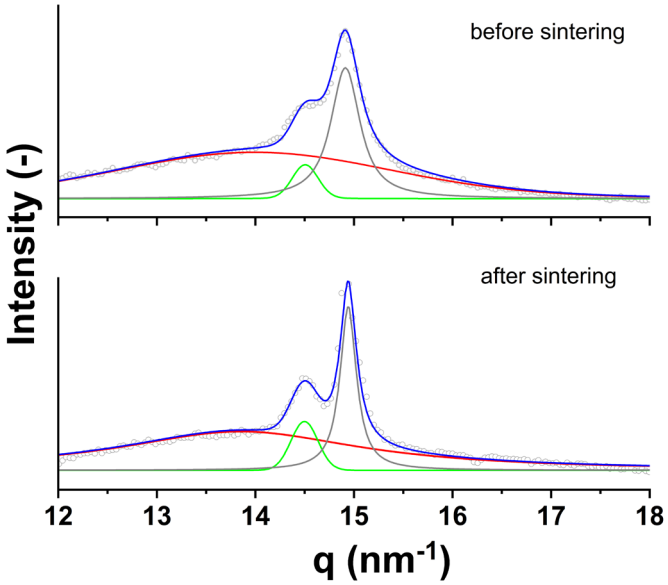

Figure 4: Integrated intensity as a function of scattering vector q, before and after sintering for 200 µm diameter PA12 particles with 2 wt% CNTs.

We successfully demonstrated the capability of the setup to perform in-situ experiments. Future experiments at different processing conditions for different classes of polymers, like high performance polymers, e.g. 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. During the present set of experiments, we could clearly show the effects of CNTs on the crystallisation of laser sintered polymers. To further analyse these effects, we will perform

additional characterizations such as differential scanning calorimetry, transmission electron microscopy and rheology. By combining the information from these complementary techniques we will be able to develop a good understanding of laser sintering of polymer nanocomposites.

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

yes

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. All the experimental data collected at BM26-B will be used to understand the influence of additives in laser sintering. At present we are working on complementary characterizations of the laser sintering and subsequently we will prepare a manuscript to be submitted to an international peer-reviewed journal.

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 : 26-02-893

Experiment dates : 03/12/2018 to 07/12/2018

Participants (max 3 persons):

Name : Prakhyat Hejmady

Name : Jessica Pepe

Name : Stan Looijmans

Payment details

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

Name: TECHNISCHE UNIVERSITEIT EINDHOVEN

City: Eindhoven