

DUBBLE – EXPERIMENT REPORT

We kindly request you to answer the questions (max 2 pages) and return the form to NWO **within 2 months of the completion of the experiment** to dubble@nwo.nl

Beam time number: 26-02-706		File number: 35698
Beamline: BM26-B	Date(s) of experiment: 05/11/2014-06/11/2014	Date of report:
Shifts: 6	Local contact(s): G. Portale	

1. Who took part in the experiments? (Please indicate names and affiliations)

Harm Caelers¹

Emanuele Parodi¹

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

2. Were you able to execute the planned experiments?

NO. Due to the problems explained later (in question 3) only a small part of the experiments could be executed. In the end the experiments could start on the second day of our 2-day beamtime in the evening at 18.00 hour. So we could only measure in the last night. Therefore we decided to do only fast experiments. Furthermore because of the orientation WAXD was not combined with SAXS, but instead WAXD patterns were recorded from compression tests performed at elevated temperatures, using a custom made oven.

3. Did you encounter experimental problems?

YES. In the night from 4 to 5 November a water cut in the ESRF took place. Therefore the turbo pump could not be cooled any more, and as a result it stopped working. The vacuum on the monochromator vented away, resulting into problems. An example of the problems was that the beam started to drift. This also caused huge deviations in the intensity measured at the beamstop, also during the experiments we were able to do.

4. Was the local support adequate?

YES. The problem with the monochromator was complicated, but in the end G. Portale and D. Detollenaere managed to solve the problems. The setup was mounted by F. Ledrappier and the result is shown in the picture below on the left. The custom made oven is shown on the right.



Fig. 1: Left, the experimental setup with Zwick Z5.0 tensile test machine in the hutch of DUBBLE. Right, the custom made oven.

For more information please contact the secretariat, tel.: +31-70-3494011, e-mail: dubble@nwo.nl

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

YES. We are able to see phase transitions from iPP gamma phase to alpha phase at elevated temperatures (110 degrees Celsius) as a result of the applied deformation. Also in case of beta crystals we see a transformation towards alpha crystals. The alpha crystals remain in the alpha phase however, also in case of alpha crystals a decrease in crystallinity is observed. This is shown in the figures below, where the results for all three crystallographic structures, measured at 110 degrees and a strain rate of 0.01 reciprocal seconds are shown. Also the true stress resulting from the applied true strain, as a function of time is shown here. Similar results are obtained for measurements performed at 50 degrees Celsius and room temperature. In that case the crystallinity dropped upon the application of strain

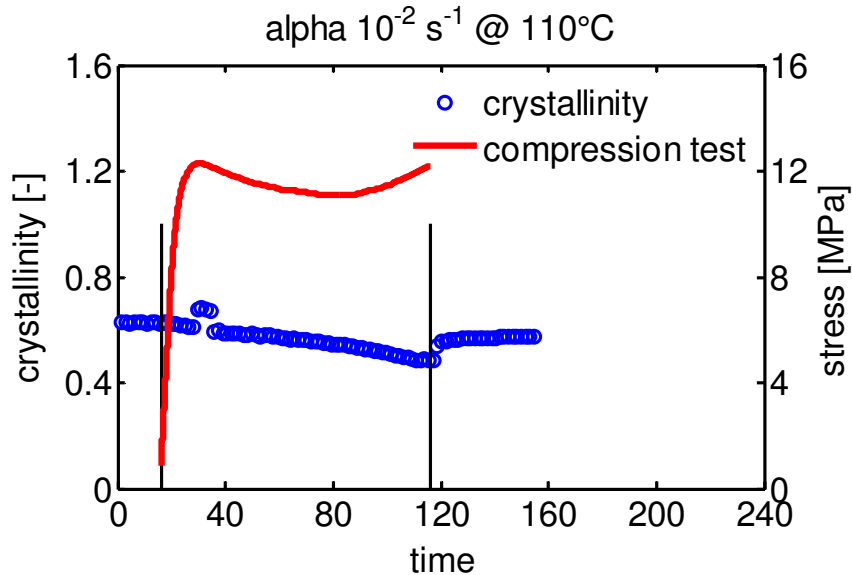


Fig.2: Crystallinity and true stress as a function of time, measured at 110 degrees Celsius and a strain rate of 0.01 reciprocal seconds on the alpha samples. Here no phase transition was observed.

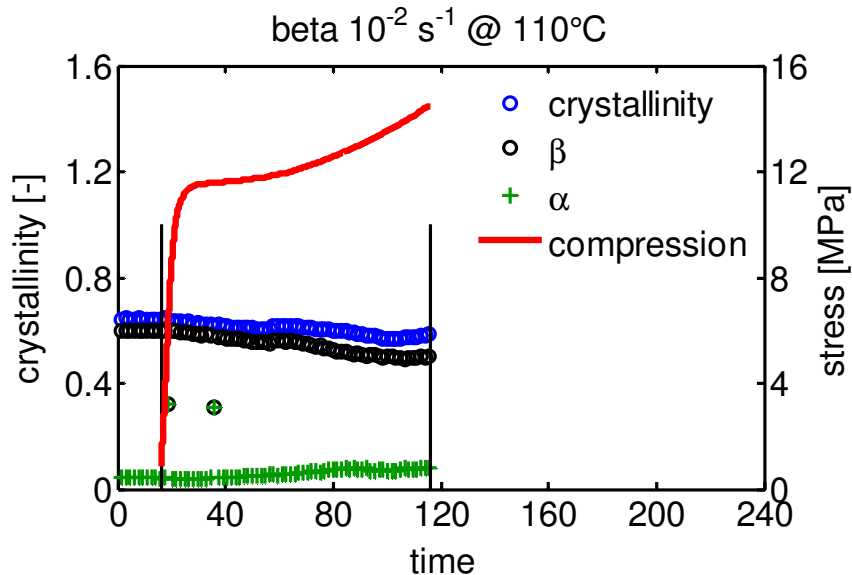


Fig.3: Crystallinity and true stress as a function of time, measured at 110 degrees Celsius and a strain rate of 0.01 reciprocal seconds on the beta samples. Here a solid state phase transition from beta to alpha crystals was observed.

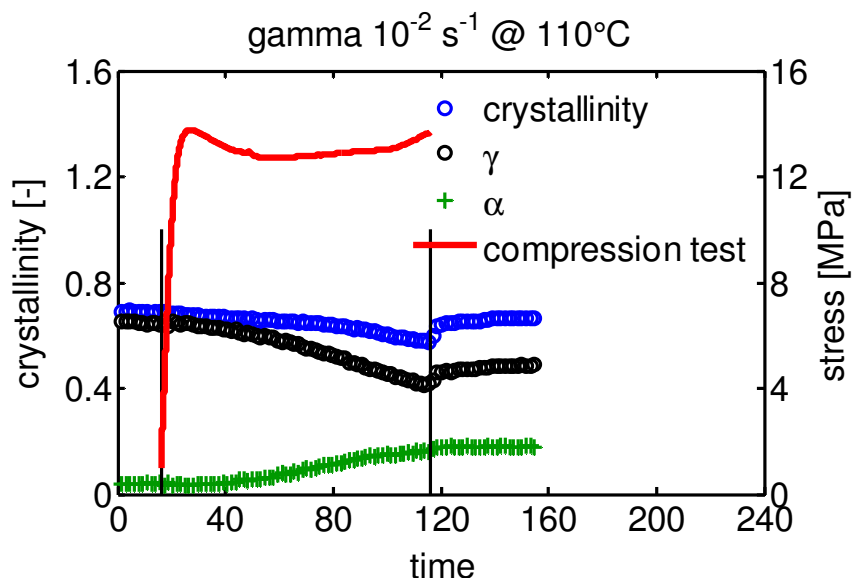


Fig.4: Crystallinity and true stress as a function of time, measured at 110 degrees Celsius and a strain rate of 0.01 reciprocal seconds on the gamma samples. Here a solid state phase transition from gamma to alpha crystals was observed.

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

YES. I would like to finish this set of experiments. I would also like to extend the data set with SAXS data.

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

NO.

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

YES. The experiments performed can be used in a work in which deformation kinetics of the different crystalline structures are investigated, as well as the failure kinetics. Although the set of data is not complete yet, it can help us to understand and interpret the observed failure behaviour. The submission can hopefully take place in 2016.

9. Additional remarks

NO.