ESRF	**Experiment title:** Mechanisms of plastic deformation and structural damage of bulk polyamide 6 : influence of molecular weight, initial microstructure and experimental conditions	**Experiment number**: SC 3758
Beamline: BM02	**Date of experiment**: From : 17 Nov 2013 (8h00) to : 19 Nov 2013 (8h00)	**Date of report**: 15 July 2014
Shifts: 6	**Local contact(s)**: Cyrille Rochas	*Received at ESRF:*

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Report:

The initial proposal of the in situ mechanism of plastic deformation and strain-induced damage in bulk PA6 on the BM02 beamline involved Wide-Angle (WAXS) and Small-Angle X-ray Scattering (SAXS) experiments both below and above the temperature of the glass transition of the amorphous component of the semi-crystalline PA6. The 12 shifts proposal was finally accepted for 6 shifts, so that only a part of the project could be performed.

During this 2-days experiment WAXS and SAXS experiments on PA6 have been performed at 120°C, i.e. above the glass transition. Two PA6 samples of molecular weight 15 kg/mol and 30 kg/mol have been studied. Moreover, various crystallization procedures were applied to these materials in order to generate different morphologies : the crystal lamellae thickness was varied in the range 7-12 nm associated with a weight fraction crystallinity in the range 25-35 %.

WAXS revealed different crystalline structures for the various PA6 samples : the more stable monoclinic form was observed for isothermal crystallization or high temperature annealing whereas the metastable mesophase and hexagonal form co-existed for injection-molded samples. Different deformation mechanism were observed : the lower the crystallinity and crystal thickness the more homogenous and uniform the crystallographic orientation behavior. High crystallinity samples displayed double texturing indicating a complex deformation mechanisms : it seems that in a 1st stage the very stiff crystalline lamellae are tilting towards to draw direction before to break into fragments that undergo chain unfolding. Moreover, in the case of initially rapidly cooled samples, strain-induced phase transition clearly appeared beyond the yield point (occurrence of plastic instability). Detailed analysis of the processes in still in progress in both situations.

SAXS revealed very little cavitation under tensile drawing at 120°C. Moreover SAXS allowed following the structural transformation of the spherulitic lamellar morphology into fibrillar structure in parallel with the crystallographic orientation and crystalline phase transformation. It is worth noticing that in the very recent proposal 02-01-847 (July 9-13/2014) WAXS and SAXS experiments have been carried out below the glass transition temperature, namely -10°C that is the temperature at which PA6 displays the minimum visco-elastic energy dissipation. Cavitation was clearly observed in such conditions. Work is in progress to analyses and combine data from the two proposals.

This work makes part of the PhD thesis of C. Millot, MATEIS-INSA de Lyon. The PhD defense is scheduled for Novembre 2014. Two papers are in preparation as a result of these experiments. An oral presentation regarding the deformation mechanims of PA6 assessed from the in situ SAXS experiments has been given at the DEPOS25 conference in Presqu'ile de Gien, March 2014.