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ESRF

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office via the User Portal:

https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

Reports supporting requests for additional beam time

Reports can be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title: Origin of the ductile behaviour of PLA/Talc nanocomposites induced by the biaxial texturing**	**Experiment number**: 02-01-857
Beamline: BM02	**Date of experiment**: From : 07/05/2015 to : 10/05/2015	**Date of report**: 09/12/15
Shifts: 8	**Local contact(s)**: C. Rochas	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

This study is aimed at characterizing the origin of the ductile behaviour induced by the biaxial stretching process of PLA-Talc nanocomposites. While PLA and its nanocomposites intrinsically exhibit a brittle behaviour, recent experiments carried out in our laboratory highlighted a significant increase of the elongation at break leading to a ductile behaviour upon stretching at room temperature of these systems when biaxially stretched. This property modification, interesting for packaging application, remains unexplained. Besides, a transition between the deformation mechanisms from crazing to shear banding is suspected. Consequently this work, which combines WAXS and SAXS experiments, is aimed at i) confirming that ductility of bi-axially stretched PLA arises from a transition of the plastic deformation mechanisms and ii) determining the structural origin of the improvement of the elongation at break observed for neat PLA and PLA/Talc nanocomposites even at high talc loadings.

Note : in this report the following notations where used:

- *PLA for the unfilled material*
- *PLA/XT for the composites with X being the clay weight content*

The first part of the study was devoted to the investigation of the influence of talc on the mechanical behavior of the materials. A particular attention was paid to the study of the influence of the talc content on the crazing formation kinectis as well as its influence on the craze microstructure. A summary of the results dealing with this point are depicted in figure 1.

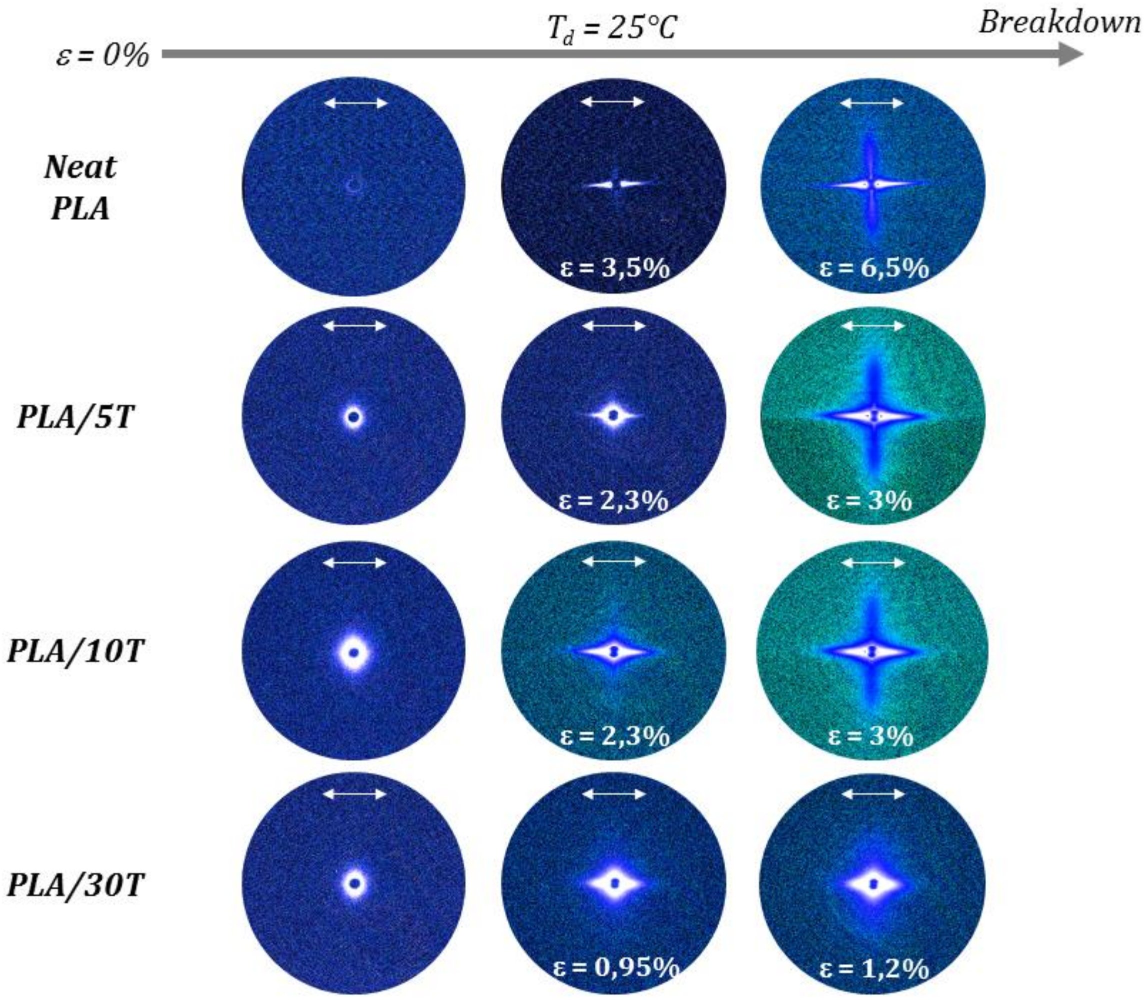

Figure 1. SAXS patterns recorded before stretching and during stretching at crazing activation and before sample's break for the PLA nanocomposites filled with different clay contents.

As can be seen no central scattering is observed for unfilled PLA while an intense central isotropic signal, arising from the clay scattering, is observed for the nanocomposites.
From the experiments carried out it was pointed out that :

- The increase of the Talc content involves an earlier activation of the crazing mechanism. This behavior is attributed to the local stress concentrations involved by the presence of the clays and explain the fact that the increase of the clay content decreases the stretchability of the material.
- The SAXS patterns recorded at sample's break differ depending on the Talc content indicating differences in the crazes microstructure. Partcularly, the analyses of the

integrated intenisty profiles have shown that the presence of talc platelets leads to the formation of wider crazes and that the microfibrils into the crazes are less organized.

The second part of the study was aimed at understanding the origin of the ductile behavior of previously biaxially stretched samples. The results obtained in the case of PLA and PLA/10T, unstretched and bi-axially stretched at T = 70°C up to λxλ = 2x2 are depicted in figure 2.

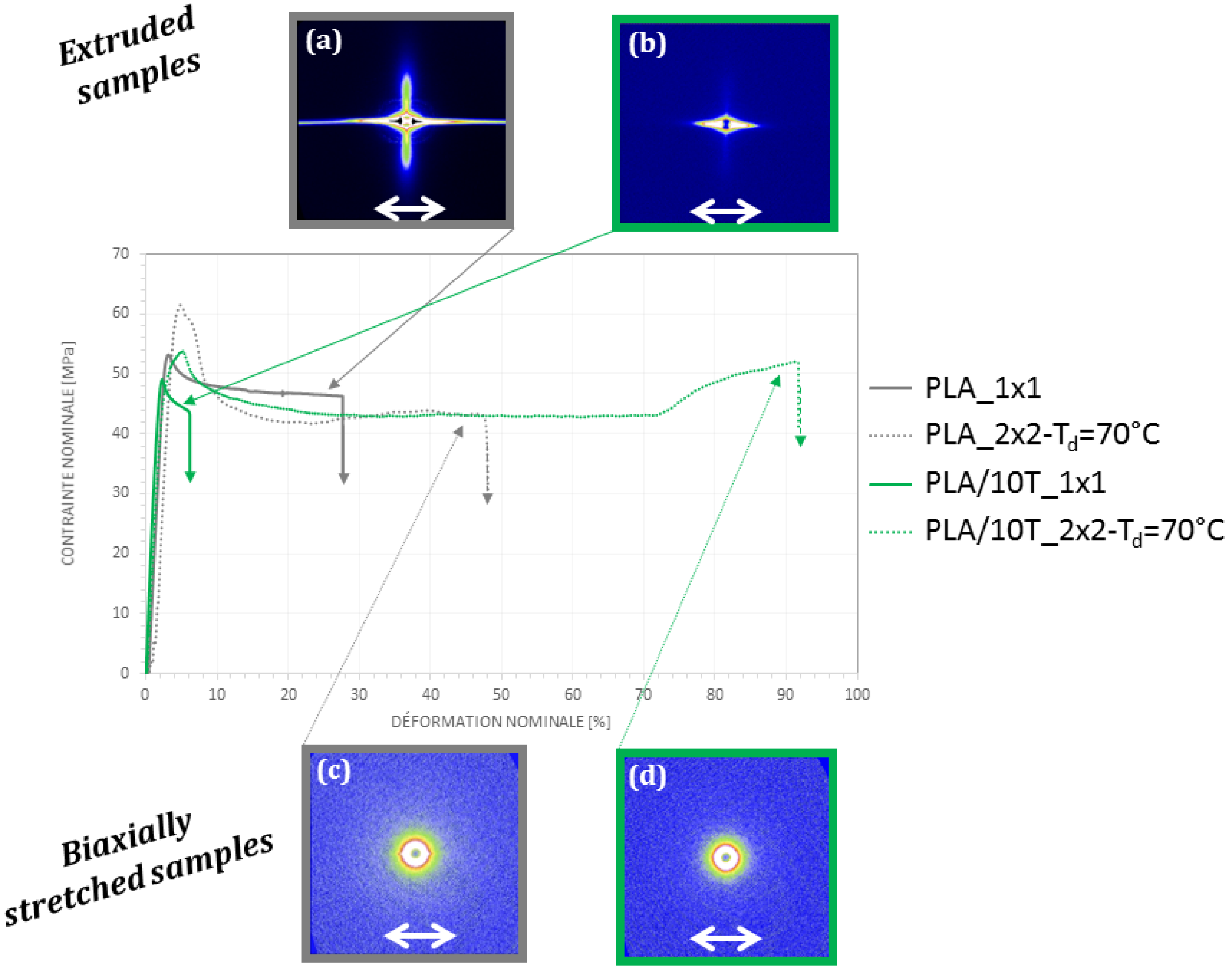

Figure 2. SAXS patterns recorded defore sample's break for PLA and PLA/10T samples unstretched or biaxially stretched at T = 70°C up to λxλ = 2x2.

As can be seen, the unstrecthed samples both exhibit a classical behavior with the activation of the crazing mechanism, responsible of the brittle behavior observed. On the other hand, previously biaxially stretched samples exhibit fairly larger elongations at break and the SAXS patterns don't show any trace of the activation of the crazing mechanism.

This result, encoutered for all the samples, clearly shows that the ductile behavior observed originates from the inhibition of the crazing mechanism during drawing. In other words this means that biaxial stretching has a positive effect on the stretchability of PLA, whose main drawback is indeed its fragility.

The influence of the biaxial stretching conditions, i.e. biaxial stretching temperature and elongation ratio, were investigated. Besides it was found that the optimal conditions, leading to the subsequent higher elongation at break were a stretching temperature of 70°C up to a draw ration of λxλ = 2x2.

Finally the origin of the crazing inhibition phenomenon was also assessed. To that the structural characterization of the biaxially stretched materials was carried out by means of WAXS. Some of the results obtained are depicted in figure 3.

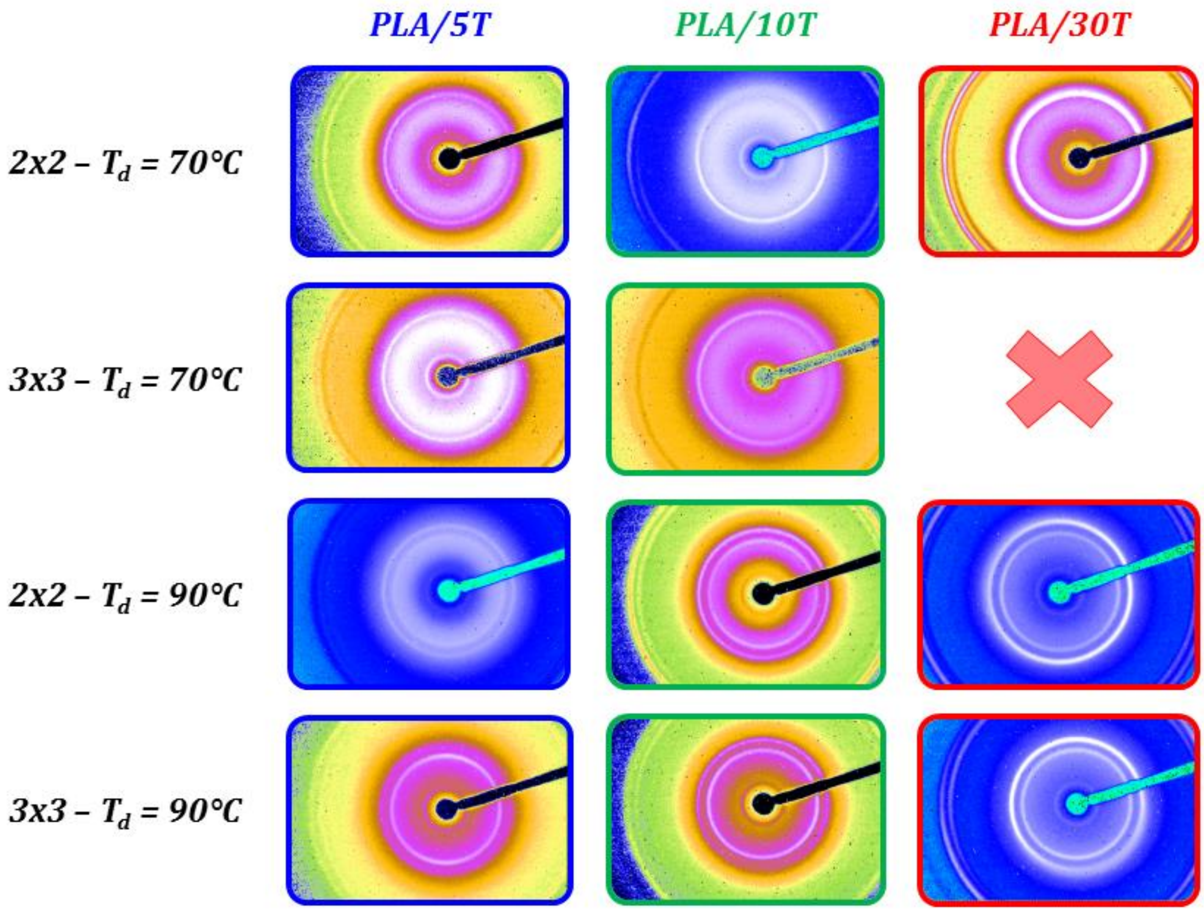

Figure 3. WAXS patterns of samples biaxially stretched under different conditions.

As can be seen, various structures varying from a fully amorphous sample to a semi-crystalline one can be induced upon biaxial stretching depending on the conditions. Taking into account that all the samples exhibit a ductile behavior after being biaxially stretched, it can be conclude that it is not the presence of a crystals which inhibits the crazing mechanism.

Consequently it is assumed that crazing inhibition rather originates from macromolecular orientations aspects. Analyses for quantifying the macromolecular orientation depending on the biaxial stretching condition as well as modeling studies of the mechanical behavior are under progress in order to assess this point.