ESRF	**Experiment title:** Time-resolved studies of thermal welding of polymer fibers	**Experiment number**: **MA- 664**
Beamline: ID02	**Date of experiment**: from: 11 February 2009 to: 13 February 2009	**Date of report**: 01.03.10
Shifts: 6	**Local contact(s)**: Peter Boesecke	*Received at ESRF:* *Guillaume Colombe* *Matthieu Defaux* *Denis Anokhin* *Martin Rosenthal*

Names and affiliations of applicants (* indicates experimentalists):

G. Colombe[1*], D.A. Ivanov[1]

[1] **Institut de Sciences des Matériaux de Mulhouse, LRC CNRS 7228 - UHA, 15, rue Jean Starcky, BP 2488, 68057 Mulhouse CEDEX, France.**

Report:

In the recent years, applications of nonwoven fiber fabric products have been extensively grown. This is particularly the case of biomedical and healthcare fields. The contact of fibers with a pre-heated profiled surface of the calander allows consolidating the fiber mat by locally welding the fibers onto each other. The mechanical performance of nonwoven materials depends on the mechanical properties of the constitutive fibers, the anisotropy of the fiber network and the welded bonds between the fibers giving the ability of transferring stress within the fiber network[1-3].

During the thermal bonding process, the properties and structure of fibers such as the type of the crystalline polymorph and chain orientation can be significantly modified. To understand the impact of the welding process and the accompanying miro-structural changes occurring in the welding spots on the mechanical stability of the web[4-6] one has to explore in detail the mechanisms of welding. In particular, it is important to address the process of melting and the following solidification using SAXS/WAXS with a sufficient detection rate.

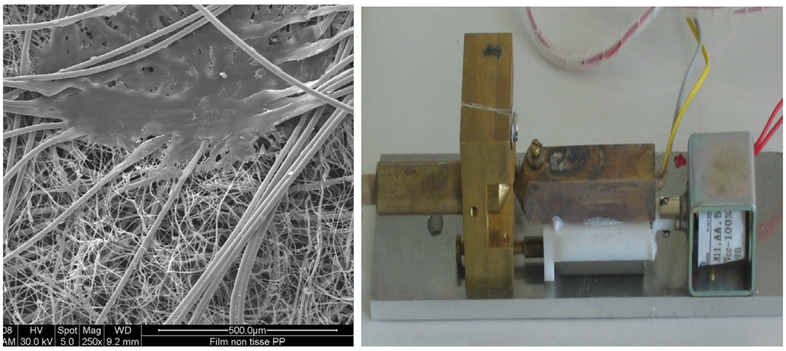

Fig.1. a) Scanning electron micrograph of a welding spot in a nonwoven fabric of isotactic polypropylene (left), home-made welding machine (right).

In this perspective, we have designed a laboratory prototype of a welding machine in order to be able to study the process with X-ray scattering. In the industrial bonding process, the fibres are deposited on a thread mill, and the bonding process occurs when the fibres pass through the two calandars, one of which has metal needles assembled on a conventional metal cylinder. The needles are heated to an elevated temperature ensuring the welding to happen within the contact time with the fiber mat (approx. 1 ms).

In our system, instead of a needle and flat support we decided to use two anvils, because the flat part might mask the X-ray intensity scattered at wide angles. In our prototype, one anvil is heated to the temperature of the bonding system, while the second one is preset at a somewhat lower temperature. The anvil corresponding to the flat part is fixed, the other anvil can be moved. The motion of the anvil is controlled by an electro-magnet, in order to get quick and controlled motion. The experiments have been carried out on a variety of melt-spun fibers of isotactic polypropylene (iPP) exhibiting very different microstructure and orientation.

Based on the obtained time-resolved 2D WAXS/SAXS patterns, one can examine in some detail the evolution of the sample micro-structure during the anvil punch. Figures 2 and 3 exemplify the microstructural evolution for the iPP fibers containing essentially the so-called smectic mesophase. At the beginning, the WAXS patterns exhibit two broad diffraction peaks along the equatorial direction (Fig. 2). In the SAXS region, the interference maximum displays a significant azimuthal spread (Fig. 3). During the anvil punch, the smectic mesophase rapidly transforms in the crystalline alpha-phase, which shows a series of sharp equatorial reflections in WAXS. This transformation happens before the sample has reached the target temperature of 140°C, as the crystalline peak positions continue moving to smaller angles with time due to the sample heating. In the SAXS patterns, the initial interference maximum becomes more concentrated on the meridional direction, which indicates on significant improvement of the chain orientation. At the same time, the radial position of the maximum continuously shifts toward smaller angles, as can be visualized in figure 3. Thus, the third SAXS pattern from the left corresponds to the moment when the sample has reached the thermal equilibrium. After the anvil retraction, as the sample gets cooled down to room temperature, the crystalline peaks move back, and the degree of crystallinity increases.

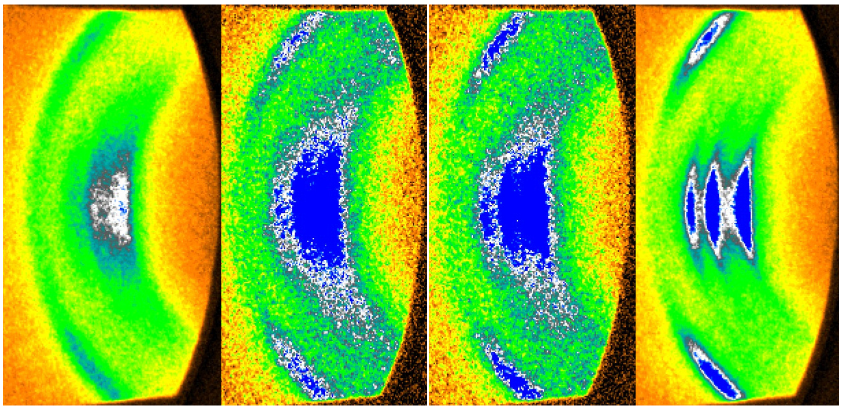

Fig.2. From left to right: 2D WAXS patterns corresponding to a fibre bundle before, during and after punch at 140°C, respectively. The time lapse between successive images is 100 ms.

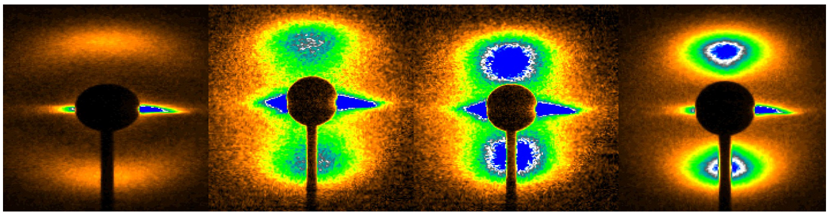

Fig.3: From left to right: 2D SAXS patterns corresponding to a fibre bundle before, during and after punch at 140°C (cf. the caption of figure 2).

Concluding, the conducted time-resolved experiments on thermal welding allowed understanding the correlation between the temperature of the anvil and the final state of the structure. The sequence and nature of the thermal events observed in these experiments such as the structural transition from the smectic mesophase to the stable crystalline phase before welding can open new perspectives in fabrication of nonwoven fabric from fibers of isotactic polypropylene.

References:

1. Nanjundappa, R. ; Bhat, G. S.; *J. Appl. Pol. Sc.* **2005**, *98*, 2355.
2. Baht, G.S.; Nanjundappa, R. *Thermochemica Acta* **2002**, 323.
3. Baht, G.S.; Chand, S. *Thermochemica Acta* **2001,** 155.
4. Wei, Q.; Liu, Y. *Polymer testing*, **2007**, *26*, 2.
5. Park, H-J.; Na C-K. *J. Coll. Interf. Sci.*, **2006**, *301*, 46.
6. Wei, Q.; Li, Q. *PolymerTesting*, **2006**, *25*, 717.