



	<b>Experiment title:</b> Scanning X-ray microscopy for polymer applications	<b>Experiment number:</b> SC-770
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<b>Shifts:</b> 12	<b>Local contact(s):</b> S. Roth	<i>Received at ESRF:</i>
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**Report:** Reaction injection moulding offers many opportunities to compatibilise different polymers by combining polymer segments of the blended polymers with the use of chemical compounds like copolymers, which can act as a bridge. New PTFE-polyamide compounds produced by reactive extrusion methods show very good mechanical and tribological properties combined with excellent wear resistance [1]. We are interested to look for failure mechanisms if these innovative materials were exposed to typical conditions of application (i.e. shear or tensile stress, respectively).

The present study focuses on the investigation of PA6 and PTFE blends prepared from PA6 and electron irradiation treated PTFE with reactive end groups. The final product properties depend on the virgin polymer properties (i.e. molar mass, viscosity, processing temperatures, crystallinity, etc.) as well as the interface between the combined polymer partners. The investigations are focused on two experiments:

**1<sup>st</sup>** - The characterisation of the **molecular and morphological structure induced by the application of tensile stress** by means of scanning microfocus X-ray scattering techniques. Samples that were a priori stretched under two different rates and brought to fracture and afterwards investigated with  $\mu$ -WAXS and  $\mu$ -SAXS using small steps. Pure PA6 specimens were also prepared using the same protocol for reasons of comparison. WAXS results for the pure PA6 reveal that there is a change in crystallinity prior to and after stretching. This change is reflected by the alteration of the ratio between the  $\alpha$ - and  $\gamma$ -modifications of PA6. Furthermore, the examination of the 2D-WAXS patterns reveals stress-induced distortion of the crystal unit cell. There is also a notable tilting of the crystals along the direction of the applied stress, which is more pronounced close to the final break zone. This may reflect the mode of failure of the material, which usually consists of extensive fibrillation at the fractured surface. 2D-SAXS patterns reveals that there is crazing in the material, and that the crazes are all located normal to the direction of the applied stress. In addition, the SAXS results indicate that there is tilting also on the micro-crystallite level (lamellae) with the tilting angles being in the same order of magnitude with the WAXS results for the crystals. Finally, WAXS scans (Fig.1) show also the existence of an outer skin of the order of 100  $\mu$ m at each side of the samples with different morphological characteristics from the bulk (core) caused by injection-moulding of the specimens.

2<sup>nd</sup> - The characterisation of the **crack propagation and structural changes in the near crack region** of notched specimens by means of the same techniques. For this reason, the samples were prepared in a special manner and a notch was introduced with the use of a sharp wedge. Subsequently, the samples were brought on the X-ray apparatus for post mortem examination of the notch induced crack region. Preliminary results for the pure PA6 show that a crack has been propagated for about 5.2 mm from the end of the notch, and in a slight angle from the horizontal direction (direction of the notch). WAXS and SAXS 2D-patterns (Fig. 2) reveal that there is significant deformation of the material at the vicinity of the crack boundaries. Additionally, in this region we observe alterations of the ratio between the  $\alpha$ - and  $\gamma$ -modifications of PA6 as well as local changes in both the crystal and lamellae orientation. The scans from the area close to the tip of the crack reveal the existence of some micro-crazes at the end of the crack into the bulk material. The regions influenced by the wedge penetration do not transcend above 300  $\mu\text{m}$  from the nick. Furthermore, the regions influenced by the crack are located in an area of no more than 200  $\mu\text{m}$  around the crack (laterally).

Future analysis of the results (currently in progress) for the PA6/PTFE blends is expected to reveal significant differences in the materials behaviour (phase transitions, orientation changes), as well as in the crack propagation and in the zone close to the crack tip, due to the differences in the morphological features between the pure PA6 and the PA6/PTFE blends. This is due with the phase separation and blend compatibilisation of the PA6/PTFE compounds [2].

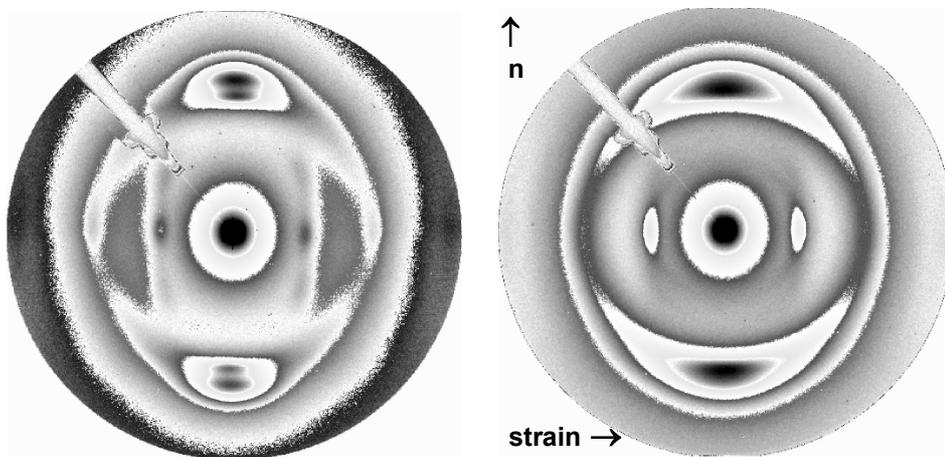


Fig. 1: Representative WAXS 2D-patterns of a stretched dumb-bell of PA6 (left: skin - dominated by  $\alpha$ -modification, right: core - dominated by  $\gamma$ ; n: normal direction of specimen, strain: stress direction)

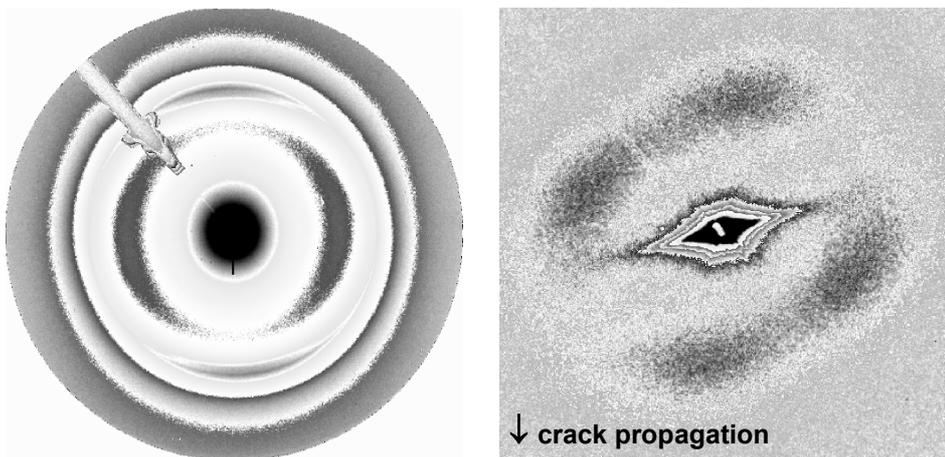


Fig. 2: Representative WAXS and SAXS 2D-patterns of PA6 in the near crack region (zone of strongest deformation: primary macroscopic crack end)

## References

- [1] Lehmann, D., Hupfer, B., Lappan, U., Pompe, G., Häußler, L., Jehnichen, D., Janke, A., Geißler, U., Reinhardt, G., Lunkwitz, K.: New PTFE polyamide compounds, *Designed Monomers and Polymers* (accepted 12/2001)
- [2] Jehnichen, D., Pohlers, A., Zafeiropoulos, N.E., Kummer, S., Roth, S., Burghammer, M., Schneider, K., Lehmann, D., Stamm, M.: ( $\mu$ -SAXS and  $\mu$ -WAXS investigations on stretched samples of reactive compatibilised PA6/PTFE materials produced by injection moulding); (The use of synchrotron radiation for polymer microscopy applications: Investigation of notch induced crack propagation by use of  $\mu$ -SAXS and  $\mu$ -WAXS in PA6/PTFE materials produced by injection moulding), (working titles), *High Performance Polymers* (in prep.)