<b>ESRF</b>	<b>Experiment title:</b> Microstructure of the interface in isotactic polypropylene composites	Experiment number: SC-838
Beamline: 1D13	<b>Date of experiment</b> : from: 14 of february 2001 to: 17 of February of 2001	<b>Date of report</b> : 16 of august 2007
Shifts: 9	<b>Local contact(s)</b> : Dr. Christian RIEKEL	Received at ESRF:

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## **Report:**

The results of this experiment have been published in:

J. Torre, M. Cortázar, M.A. Gómez, G. Ellis, C. Marco, C.Riekel, P. Dumas Nature of the crystalline interphase in sheared iPP / Vectra fiber model composites by microfocus X-ray diffraction and IR microspectroscopy using synchrotron radiation. **Macromolecules 39 (16), 5564-5568, 2006.** 

The conclusions from the experiment are:

We have studied iPP matrices with sheared LCP-fibres by thermo-optical microscopy with polarised light, IR microspectroscopy and X-ray microdiffraction employing synchrotron radiation sources. We have obtained spatially resolved crystallographic information for the first time from the polymorphic polymer – LCP fiber interphase region, which have allowed us to unequivocally relate spectroscopic and structural evidence to morphological details. The occurrence of a thin  $\alpha$ -phase close a sheared fibre, which generates the cylindritic  $\beta$ -phase has been clearly demonstrated by two structure-sensitive techniques.

The complimentarity of the synchrotron IR microspectroscopy and X-ray microdiffraction techniques in terms of sample preparation and spatial contrast provide a powerful method for

the analysis of complex interphase behavior in reinforced polymer composites. The combined use of these techniques can be particularly important when studying the structure of semicrystalline polymer composites and alloys in general, since IR provides information on chemical structure, conformation and orientation, and can be used to clearly define specific areas of interest in multiphase systems, whereas X-ray microdiffraction provides precise information on the crystallisable part of the material. Finally, due to the unique characteristics of the synchrotron IR beam, synchrotron IR microspectroscopy can be performed in an open laboratory environment, and sample optimization and measurement is quite versatile facilitating the use of dual sampling strategies which allow microscopic samples to be studied in the same position in multiple techniques.

The microfocus X-ray diffraction experiments were carried out at the European Synchrotron Radiation Facility (ESRF) microfocus beam line (ID13) using a wavelength  $\lambda = 0.0948$  nm (Si-111 monochromator) and a 3  $\mu$ m diameter beam. The infrared micospectroscopy experiments were performed at the U10B beamline of the National Synchrotron Light Source at Brookhaven National Laboratory, NY, USA.