ESRF	**Experiment title:** Investigation on poly(3-hydroxybutyrate) spherulites in the pure state and in blends with cellulose tributyrate by means of microfocus x-ray diffraction.	**Experiment number:** CH-595
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Report:

Poly(3-hydroxybutyrate) (PHB) is a highly crystalline natural polyester produced by microorganisms. Its very large spherulites, if cristallized in suitable conditions, show wide extinction bands (alternative white and black, with total width up to 160 μm) when viewed in the polarized optical microscope (Fig. 1). Previous investigations carried out by X-ray diffraction showed that the *a*-axis of the ortorombic cell is radial in the spherulite, but the actual orientation of the other two axes is still a matter of debate.
The first aim of the present experiment was to determine the orientation of the *b*- and *c*- axes inside each band of pure PHB and to correlate morphological investigation with structural evidences.

Isothermal crystallization studies on blends of PHB with cellulose-tributyrate (CTB) have shown that the polymers are miscible and that the radial growth rate of PHB spherulites is affected by blend composition. The investigation of the abundance and radial distribution of CTB crystallites within spherulites of PHB/CTB blends, was the second goal of this experiment.

The experiment was carried out on samples isothermally crystallized between two cover glass in the hot stage of an optical microscope. The samples (circles of about 5 mm in diameter, 20-50μm thick) were mounted on TEM grids and aligned normally to the x-ray beam with the help of a trigonometric head. An x-ray beam of 3μm in diameter at the collimator exit was used to perform both radial scanning along the spherulite radius and area scanning of selected portion of the spherulites. In both cases a step of 3μm was used.

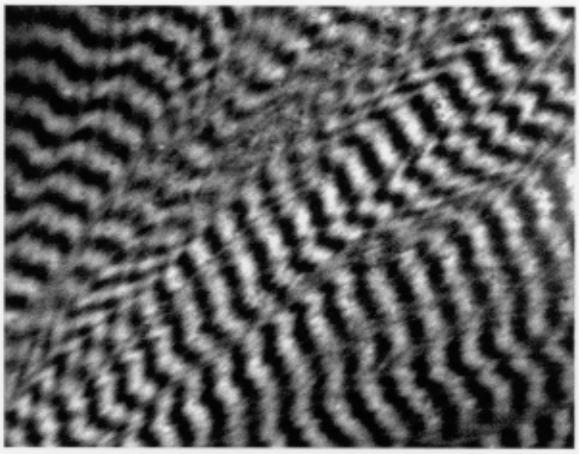

Fig 1. Optical micrograph, between crossed polars, of a portion of a PHB spherulite

From the comparisons of the single diffraction patterns obtained focusing the center of the white and black section of a band (Fig. 2 a, b) a different orientation of the PHB unit cell is evident. The data collected in this experiments are presently under processing, in order to verify the hypothesis that banding originates from periodic torsion of the lamellar crystals during growth.

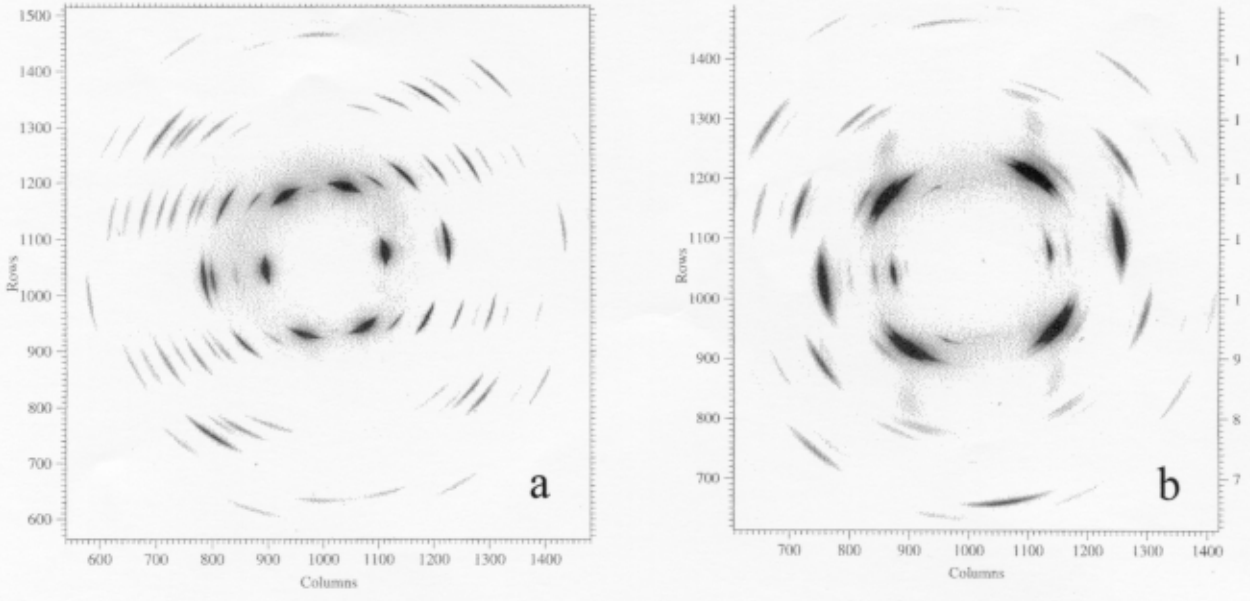

Fig 2. WAXS pattern of pure PHB

As regards the second system investigated, from the preliminary PHB/CTB blends data processing, it appears that the composition ratio along the radius of the spherulites it is almost constant.

Additional experiments will be necessary to contribute to a better understanding of the banding process in PHB spherulites. In particular, it would be useful to investigate structural details of PHB banded spherulites in the presence of a second miscible amorphous component, according to a proposal presently submitted to ESRF.