Ciencias Químicas Benemérita Universidad Autónoma de Puebla, Mex. <sup>b</sup>Maestría en Dispositivos Semiconductores ICUAP, Puebla, Pue. Mex. <sup>c</sup>Depto. de Ing. Eléctrica-SEES, CINVESTAV-IPN, Av. IPN # 2508, Apdo. Postal 14-740, 07300 México 14 D. F., Mex. E-mail: javier.martinez@icbuap.buap.mx

Thin films of cadmium sulfide (CdS) and cadmium carbonate (CdCO<sub>3</sub>) were grown onto glass substrates by means of the chemical bath (CB) method. The temperature of grown was selected between 23-80 °C. At low temperatures, CdCO<sub>3</sub> is the compound predominant in the layers, whereas at high temperatures CdS is the compound deposited on the substrate. The gradual transition from an insulator CdCO<sub>3</sub> to a semiconductor CdS growth occurs when values a mixture increases. Physical properties of films they are studied by means of X-ray diffraction, and optical absorption.

Keywords: semiconductors, diffraction, cadmium carbonate

#### P.11.08.1

Acta Cryst. (2005). A61, C391

# Scanning X-ray Scattering Study on Structural Changes at Crack Tips in PVDF

<u>Guenther A. Maier</u><sup>a</sup>, Gernot Wallner<sup>b</sup>, Reinhold W. Lang<sup>b</sup>, Peter Fratzl<sup>c</sup>, <sup>a</sup>Material Center Leoben, Institute of Metal Physics, University of Leoben, Leoben, Austria. <sup>b</sup>Polymer Competence Center Leoben and Institute of Materials Science and Testing of Plastics, University of Leoben, Leoben, Austria. <sup>c</sup>Max Planck Institute of Colloids and Interfaces, Dep. of Biomaterials, Potsdam, Germany. Email: guenther.maier@unileoben.ac.at

Scanning small angle X-ray scattering approaches have been demonstrated to provide structural information at the supra-molecular level with positional resolution in the micron range. We use this technology on a lab-system with a beam size of 0,1mm to study deformation mechanisms around crack tips in poly(vinylidene fluoride) (PVDF), a semi-crystalline polymer which is known to show deformation-induced phase transitions. Fracture in semi-crystalline polymers is accompanied by the formation of a plastic zone, consisting either of shear bands, micro cavities or crazes. The supramolecular deformation processes around the crack tip are essential for the progression of the crack and, hence, in defining the toughness of the material. Due to the enormous stress gradients around the crack tip they are difficult to assess and -in most polymers- only poorly understood. The use of position resolved scatting methods for investigations of the crack tip area provides detailed information of the structural changes during crack propagation. Our study shows a localized transformation of  $\alpha$ -PVDF into the  $\beta$ -modification near the crack tip. The β-modification is forming fibers bridging crazes and cracks and, hence, considerably contributing to the toughness of the material.

### Keywords: polymers, fracture, scanning-SAXS WAXS

#### P.11.08.2

Acta Cryst. (2005). A61, C391

#### An Investigation into the Effects of Temperature and Crystallization Conditions on the Lattice Parameters of Ultra Long n-alkane Crystals

<u>Timothy D. Lord</u><sup>a</sup>, S. Hanna<sup>b</sup>, Å. Kvick<sup>a</sup>, J.K. Hobbs<sup>c</sup>, A.E. Terry<sup>d</sup>, J. Wright<sup>a</sup>, <sup>a</sup>E.S.R.F. BP220, Grenoble Cedex, France. <sup>b</sup>Dept. Phys., Uni. Bristol., BS8 1TL, UK. <sup>c</sup>Dept Chem, Uni. Sheffield S3 7HF. UK. <sup>d</sup>Dept. Chem. Eng, Eindhoven University of Technology, PO Box 513, 5600MB Eindhoven, The Netherlands. E-mail: lord@esrf.ft

Ultra-long, strictly monodisperse n-alkanes, with a chain length between 100 and 400 carbons, crystallize into extremely regular lamellae with a thickness that is an integer fraction of the extended chain length. They have been investigated as model systems for polymer crystallisation, crystal annealing and melting.

High resolution time resolved wide angle X-ray scattering has been performed on beamline ID11 at the ESRF, in Grenoble. Changes in the lattice parameters of a range of ultra long n-alkanes have been determined as a function temperature. Emphasis has been placed on observing the transitions of chain unfolding and melting. The principal component of unit cell expansion is directed along the a axis, with little change parallel to the b and c axes, in agreement with previous studies. Subtle differences in the lattice parameters depending on the crystal thickness and the number of folds in the chain have been observed. At room temperature the number of folds has a controlling influence over the lattice parameters, with the more folded crystals having a more expanded lattice. Crystal thickness starts to play a role as the melting temperature is approached. The effect of pressure on the temperature dependance of lattice parameters in these model systems was also investigated, in the range 0-6 kbar.

Keywords: polymer, alkane, crystal refinement

## P.11.08.3

Acta Cryst. (2005). A61, C391

Deformation Process of Polymer Spherulite Observed with Microbeam-SAXS and -WAXS

Yoshinobu Nozue<sup>a</sup>, Takashi Sakurai<sup>a</sup>, Hideaki Hori<sup>a</sup>, Tatsuya Kasahara<sup>a</sup>, Noboru Yamaguchi<sup>a</sup>, Yuya Shinohara<sup>b</sup>, Yoshiyuki Amemiya<sup>b</sup>, <sup>a</sup>Sumitomo Chemical Co. Petrochemicals Research Laboratory. Sodegaura, Japan. <sup>b</sup>Department of Frontier Sciences, University of Tokyo, Kashiwa, Japan. E-mail: nozue@sc.sumitomo-chem.co.jp

In the field of polymer processing, the understanding of polymer deformation under force is very important for the design of polymer with higher performance. Especially, the deformation of polymer spherulite by drawing is the most interested phenomenon in film processes, and clarifying the deformation mechanism of spherulite will give the better direction of polymer design for films.

To observe the inhomogeneous deformation process within a spherulite, we applied the scanning microbeam-SAXS and -WAXS technique to a deformed large isotactic polypropylene(iPP) spherulite (averaged radius is around 200-300µm) by uni-axial hot drawing. By scanning various spherulites, the deformation degree of which is different, with microbeam, we found that the order of crystalline orientation in the up- and down-side area of a spherulite drawn in the horizontal direction came to lower and lamella stacking structure was broken in the initial stage of deformation and that the order of crystalline orientation and lamella structure recovered in the latter stage of deformation. On the other hand, the order of crystalline orientation and lamella stacking in the left- and right-side of a spherulite were kept in the initial stage and they were drastically changed in the latter stage of drawing.

Keywords: microbeam, SAXS WAXS, SAXS polymer

### P.11.08.4

Acta Cryst. (2005). A61, C391-C392

# Crystal Structure and Texture Refinement of Polymers from Diffraction Images

<u>Mauro Bortolotti</u>, Luca Lutterotti, Luca Fambri, *Department of Materials Engineering and Industrial Technologies, University of Trento, Italy.* E-mail: Mauro.Bortolotti@ing.unitn.it

The crystal structure of not perfectly crystallized polymers has always been difficult to refine or determine with accuracy especially when a single crystal is not available. Most of them crystallize sufficiently only when strained in fibers. Clearly in this form they are not a single crystal but not even a random powder to permit a reliable crystal structure refinement.

In the present work, we present a methodology to analyze diffraction images of polymers to obtain crystal structure, texture and microstructural information. A laboratory image plate system has been used to collect diffraction images in transmission and reflection diffraction of aligned and strained fibers of different polymers.

The images have been processed in Maud [1] and a structure refinement approach including a Rietveld Texture Analysis [2] was performed for each polymer. An energy approach has been incorporated to help the refinement strategy as well as the use of fragments. From the texture point of view the standard function method for quantitative texture analysis has been developed and successfully applied to these systems. It permits to determine with high accuracy and precision the spread of the single polymers chains