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Thin films of cadmium sulfide (CdS) and cadmium carbonate (CdCO₃) 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, CdCO₃ 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 CdCO₃ 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

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

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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 supra-molecular 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 scattering 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 α -PVDF into the β -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

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

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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 dependence of lattice parameters in these model systems was also investigated, in the range 0-6 kbar.

Keywords: polymer, alkane, crystal refinement

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

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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 200-300 μ m) 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

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Crystal Structure and Texture Refinement of Polymers from Diffraction Images

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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

in the fibers. The strain applied to the fibers has been modeled by an integrated texture-stress model inside the program as well.

[1] <http://www.ing.unitn.it/~luttero/maud> [2] Ischia G., Wenk H.-R., Lutterotti L., Berberich F., *J. Appl. Cryst.*, 2005, **38**, 377.

Keywords: structure determination, texture, polymer

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Novel Structure of 1,1'-Disubstituted Ferrocene Derivative

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Some of the 1,1'-disubstituted ferrocene derivatives (bMAF-*n*, *n* is the carbon number in the flexible spacer) show liquid crystallinity. It was reported that bMAF-5 had S-typed molecular structure [1]. The S-shaped one of the other derivative was already reported by the other workers [2]. Recently, new U-typed one was found out in bMAF-10 [3].

In this study, the crystal structure of bMAF-2 was analyzed by single crystal X-ray diffraction method. All measurements were made by Rigaku AFC-5R diffractometer with graphite monochromatized CuK α radiation. The crystal structure obtained in this study was a monoclinic with space group *C2/c*. The residual R and wR converged on 0.036 and 0.067, respectively. The feature of the structure was very unique, because the two substituent had a bent structure around a *gauche* conformation. This molecular structure was given a name to Z-shape, which was different from those of bMAF-5 (S-shape) and 10 (U-shape).

Liquid crystallinity will be discussed from structural point of view.

[1] Nakamura N., et al, *Mol. Cryst. Liq. Cryst.*, 2005, *in press*. [2] Khan A.M., et al., *Liq. Cryst.*, 1989, **5**, 285. [3] Nakamura N., et al, *Chem. Lett.*, 2004, **33**, 358.

Keywords: ferrocene compounds, liquid crystal, crystal structure

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Crystal Structure of 1,24-Dibromotetracosane

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The crystal structure of 1,24-dibromotetracosane was analyzed by single crystal X-ray diffraction method. The compound crystallized in a monoclinic system ($a = 5.482(3)\text{\AA}$, $b = 5.381(2)\text{\AA}$, $c = 43.859(2)\text{\AA}$, $\beta = 93.07(2)^\circ$, $Z = 2$) with a space group *P2₁/c*. The molecule is centrosymmetric and its skeleton has an all-*trans* conformation including both terminal Br atoms. In the crystal, the molecules form layers with a thickness of $c/2$. In the layer, the molecules inclined to the basal plane of Br atoms. The layers are arranged in a zigzag manner between the neighboring layers making a herringbone motif just like the smectic C_A structure of liquid crystals.

The molecular and crystal structures of 1,24-dibromotetracosane are similar to those of the homologs with an even number of C atoms, *vis.* 1,12-dibromododecane[1], 1,14-dibromotetradecane[2], 1,16-dibromo-hexadecane[3], 1,18-dibromooctadecane[4], 1,20-dibromoicosane[5], and 1,26-dibromohexacosane[6].

[1] Kulpe S., et al, *Cryst. Res. Technol.*, 1981, **30**, 349. [2] Uno K., et al, *Acta Cryst.*, 2003, **E59**, o708. [3] Kobayashi H., et al, *Cryst. Res. Technol.*, 1995, **30**, 275. [4] Nakamura N., et al, *Cryst. Res. Technol.*, 1993, **28**, 953. [5] Nakamura N., et al, *Acta Cryst.*, 2004, **E60**, o1408. [6] Takamizawa K., et al, *Eng. Sci. Rep. Kyushu Univ.*, 1992, **13**, 341.

Keywords: alkyl halide, crystal structure, liquid crystal

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Crystal Structure of Monosubstituted Ferrocene Derivatives

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A series of monosubstituted ferrocene derivatives, ω -[4-(4-methoxyphenoxy-carbonyl)phenoxy-carbonyl]alkyl 4-ferrocenyl benzoate (abbreviated hereafter as MPAF-*n*, *n* = 1~11, where *n* is the number of carbon atoms in the methylene unit) were prepared in our laboratory [1].

In the present study, the crystal and molecular structures of MPAF-10 were determined by X-ray diffraction method using a single crystal. All measurements were made by Rigaku AFC-5R diffractometer with graphite monochromatized CuK α radiation. The crystal structure obtained in this study was orthorhombic ($a=8.176(6)$, $b=88.253(7)$, $c=10.140(9)\text{\AA}$, $Z=8$) with space group *Pbca*. The residual R and wR converged on 0.056 and 0.128, respectively. Good of fitness is 1.000.

This compound shows liquid crystallinity. This molecule bends a little around an ester group located in the ferrocene side. The feature of the molecule is not favorable for the liquid crystalline compound. Two molecules, however, associate head to head manner making a rod-like shape. Therefore, this compound exhibits liquid crystallinity.

[1] Hanasaki T., Ueda M., Nakamura N., *Mol. Cryst. Liq. Cryst.*, 1993, **237**, 329.

Keywords: liquid crystal, ferrocene compounds, crystal structure

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Crystal Structure of a Liquid Crystalline Ferrocene Derivative

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A series of 1,1'-disubstituted ferrocene derivative, 1,1'-bis[ω -[4-(4-methoxyphenoxy-carbonyl)phenoxy]alkoxy-carbonyl]ferrocene (abbreviated hereafter as bMAF-*n*, where *n* is the number of carbon atoms in the methylene unit) was prepared, and some of them (bMAF-3,5~12) exhibited liquid crystallinity[1,2]. In this study, the crystal structures of bMAF-3 were determined by the X-ray diffraction method in order to gain an understanding of the interrelation between the crystal structure and some physical properties. The single crystals of the compounds were obtained from a solution with a mixed solvent by the slow evaporation method. The measurement was made on a Rigaku AFC-5R diffractometer with graphite monochromatized Cu-K α radiation. The calculation was performed using the *Crystal-Structure* crystallographic software package. The crystal structure was mono-clinic system ($a=5.857(4)\text{\AA}$, $b=24.105(3)\text{\AA}$, $c=14.069(4)\text{\AA}$, $\beta=93.15(4)^\circ$, $Z=2$) with space group *P2₁*. In generally, rod-like molecules has advantageous structure to show liquid crystallinity. The molecular structures of bMAF-3 could be regarded as rod-like. In fact, they have the advantage of showing liquid crystallinity, and they give rise to liquid crystallinity.

[1] Hanasaki T., Ueda M., Nakamura N., *Mol. Cryst. Liq. Cryst.*, 1994, **250**, 257. [2] Nakamura N., Mizoguchi R., Ueda M., Hanasaki T., *Mol. Cryst. Liq. Cryst.*, 1998, **312**, 127.

Keywords: liquid crystal, ferrocene, rod-like

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Layer and Molecular Orientation in Electroclinic Liquid Crystals: Dynamic μ -Diffraction

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