diameter, $\bar{D}$, the relative width $\sigma_D/\bar{D}$ of the needle diameter distribution and the total needle cross section with respect to the cross section of the fiber as a function of elongation. Since all these parameters are closely related to moments of $h_D(D)$, and because of the fact that under Mellin convolution moments are transformed in a very simple manner [6], structural parameters can be retrieved directly from the chord distribution by means of moment arithmetics.

Final result

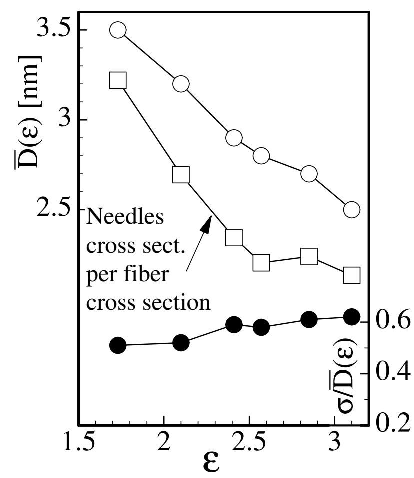

Figure 5: Structural parameters of the ensemble of soft domain needles in a PEE as a function of elongation as obtained from $g_2(x_{12})$ by moment arithmetics.

For the studied PEE and as a function of elongation ϵ Fig. 5 shows the average needle diameter, $\bar{D}$, the total needle cross section with respect to the sample cross section and the relative width $\sigma_D/\bar{D}$ of the neeedle diameter distribution. Two regions can be observed in the diagram. For elongations $\epsilon < 2.6$ the compressibility of the needles is higher than that of the surrounding matrix. Saturation effects for $\epsilon > 2.6$ indicate a hardening of the soft needles, which might be correlated to strain induced crystallization of the PTHF.

Acknowledgments

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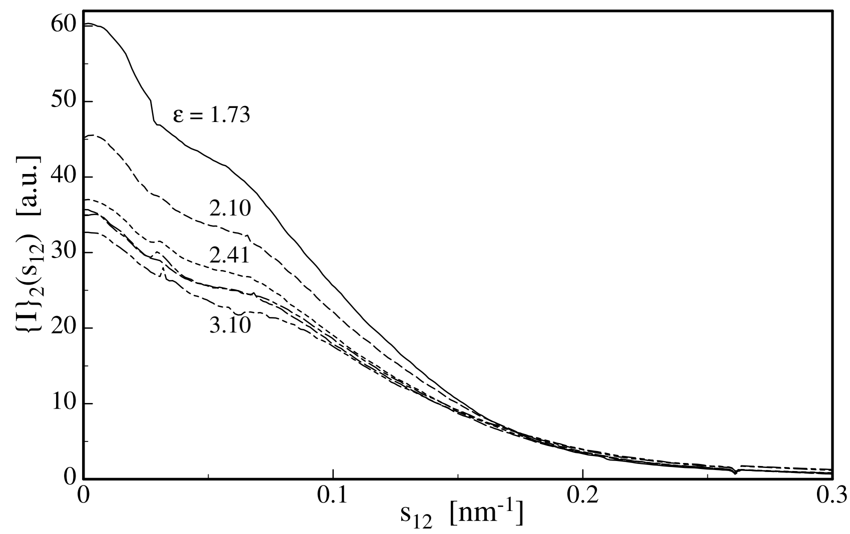

Figure 3: Projections $\{I\}_2\,(s_{12})$ of the equator scattering on to the plane normal to the straining direction as extracted from scattering patterns of a PEE as a function of elongation ϵ.

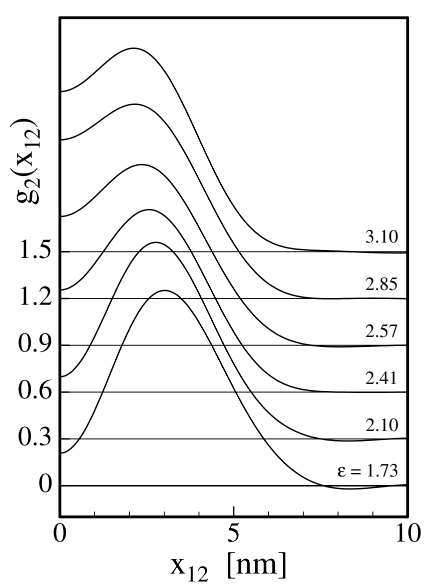

Figure 4: 2D chord distributions $g_2\,(x_{12})$ of a PEE as a function of elongation ϵ as computed from the projected equatorial scattering $\{I\}_2\,(s_{12})$.

In general $g_2\,(x_{12})$ shows the distribution of chords from needle and matrix cross sections and their correlations in the plane normal to straining direction. Experimentally only a single positive peak from uncorrelated disks is observed, indicating that in physical space the correlations among the soft domain cross sections are negligible.

Theoretical treatment

Neglecting the weak correlations among the soft domain cross sections the 2D chord distribution becomes

$$g_2\,(x_{12}) = h_D\,(x_{12}) \odot g_c\,(x_{12})\,, \tag{2}$$

where $h_D\,(x_{12})$ is the studied needle diameter distribution, $g_c\,(x_{12})$ is the chord distribution of the unit circle, and $\odot$ designates the Mellin convolution:

$$g_2\,(x) = \int_0^\infty h_D\,(y)\,g_c\left(\frac{x}{y}\right)\frac{dy}{y}. \tag{3}$$

$g_c\,(x_{12})$ is analytical. Thus the needle diameter distribution can be computed from the chord distribution. Interesting parameters are the average needle

[p. 394, left column]

Experimental

Films from Arnitel E2000/60 (by DSM, The Netherlands) were strained continuously in the synchrotron beam at beamline A2 (HASYLAB, Hamburg). Image plate detector was positioned 1.8 m behind the sample and exposed for 1 min. The material contains soft segments from polytetrahydrofurane (PTHF) and hard segments from polybutyleneterephthalate (PBT).

Observations and semi–quantitative analysis

Principal observations and peak shifts are shown in Fig. 1. During straining a two–point diagram (long period $L_{3,1}$) is observed. At an elongation $\epsilon = 1.3$ a second peak ($L_{3,2}$) emerges, the first peak vanishes behind the primary beam stop and "needle scattering" ($L_{3,n}$) appears at the equator. This observation can be explained by a microfibrillar model [4]. At low elongation microfibrils from hard and soft domains cause the two–point pattern while at high elongation hard domains are unravelled causing the formation of needle shaped soft domains which scatter about the equator.

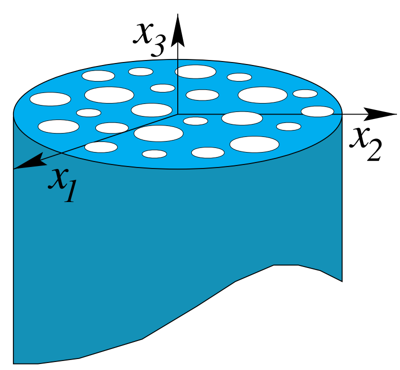

Figure 2: Sketch of the 2D fiber cross section structure, whose chord distribution is related to the projection according to eq. 1.

Projections and the transverse structure

Because of the Fourier transform relation between scattering and structure, sections in physical space are related to projections in reciprocal space. The well–known invariant, Q, is an example for a projection, which filters the non–topological parameters of the two–phase structure out of the scattering pattern. When dealing with the equatorial streak of a fiber pattern, it appears suitable to extract what Bonart called "Querstruktur" (transverse structure)[2] by computing the projection

$$\{I\}_2\,(s_{12}) = 2\int_0^\infty I\,(s_{12}, s_3)\;ds_3. \qquad (1)$$

Here $s_{12} = \sqrt{s_1^2 + s_2^2}$ and s_3 are the components of the scattering vector as indicated in Fig. 1. The magnitude of the scattering vector is defined by $|\vec{s}| = (2/\lambda)\sin\theta$, with the X–ray wave length λ and 2θ defined as the scattering angle. $\{I\}_2\,(s_{12})$ is related to a 2D two–phase system made from needle cross sections in a matrix, as indicated in Fig. 2.

Projections extracted from the image series are presented in Fig. 3. The curves show a distinct Porod region, in which the scattering falls of with s_{12}^{-3}. Small deviations are accounted to the non–ideal structure of the real two–phase system [5] and corrected accordingly, leading to the 2D interference function $G_2\,(s_{12})$ of an ideal two–phase system[6]. From the interference functions the 2D chord distributions $g_2\,(x_{12})$ (cf. Fig. 4) can be computed by 2D Fourier transformation [7].

The Equatorial Small–Angle Scattering During the Straining of Polyetheresters and its Analysis

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Introduction

Two–phase polymers with preferred orientation frequently exhibit small–angle X–ray scattering (SAXS) patterns with fiber symmetry, which can be recorded at a syncrotron beam line with high accuracy. Quite often one observes patterns with many reflections, which vary considerably as a function of tunable parameters. After a qualitative description of the observations and a semi–quantitative analysis of reflection positions as a function of the parameter values a quantitative analysis of the image series should afford profound insight into structure. The objective of the present study is the extraction of the diameter distribution describing an ensemble of oriented rodlike domains in strained material.

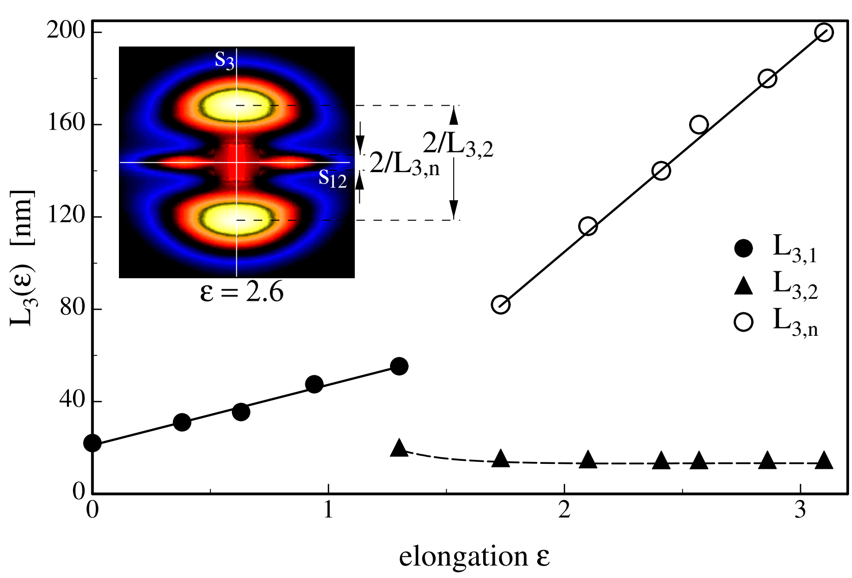

Figure 1: Principle SAXS pattern and peak shifts observed during the straining of a PEE sample. $L_{3,1}$ and $L_{3,2}$ are long periods, while $L_{3,n}$ is the reciprocal height of the form factor envelope from an ensemble of needle–shaped particles causing the equatorial scattering.

With respect to structure polyetheresters (PEE) are multiblock copolymers, whose blocks are called hard segments and soft segments respectively. As a result of phase separation a two–phase system from hard domains and soft domains is formed. With respect to material properties PEE's are thermoplastic elastomers. When strained beyond 50 %, the process becomes irreversible [1]. The relation between irreversible elongation and the materials two–phase structure is supposed to be elucidated by means of a quantitative analysis of the SAXS patterns.

Before starting to analyze, suitable reduction of the measured data should be considered. The mathematical relation between structure and scattering pattern leads to the suggestion to analyze projections of the scattering pattern [2]. In this work it is shown, how a stepwise analysis of a projection curve results in a simple model for the observed equatorial scattering. Fourier transformation into the chord distribution [3] varies the weighting of measured data in such a manner that deviations from pure "particle scattering" become negligible. Finally simple algorithms are pointed out, which allow to extract structural parameters directly from the chord distribution.

Polym. Mater. Sci. Eng. (1998), 79:393-394 [p. 393, left column]