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X-ray investigations of linear and cross-linked liquid-crystalline main chain and combined polymers

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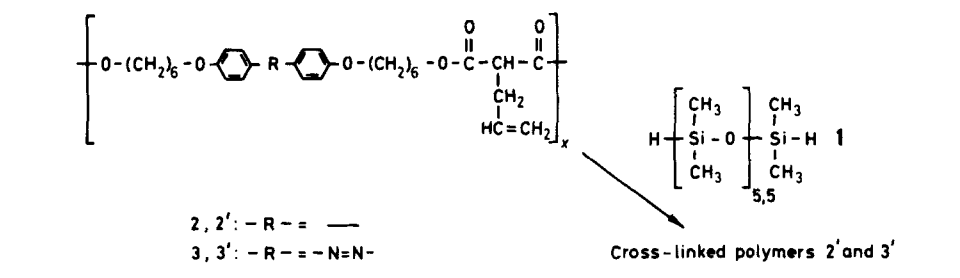
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X-ray measurements on liquid-crystalline main chain polymers and on combined polymers (polymers with the mesogenic groups in the main chain as well as in the side groups) before and after cross-linking allow the identification of the different liquid-crystalline phases. Smectic A, smectic B and probably smectic C phases are found at higher temperatures. The fibre pattern of the oriented combined polymers are analogous to the fibre pattern of the liquid-crystalline main chain polymers. The comparison implies that the mesogenic groups in main chains and side groups are oriented parallel to each other to form the liquid-crystalline phase. No hint of a microscopic bidirectional orientation is observed. Two different types of orientation were observed in the polymers on drawing, depending on the molecular weight (non-cross-linked polymers) or the elongation (cross-linked polymers). In lower molecular weight polymers the smectic layers are oriented parallel to the fibre axis on drawing. For higher molecular weight polymers and for the cross-linked polymers at higher elongation the smectic layers are oriented perpendicular to the fibre axis and the polymer chains are oriented preferentially parallel to the fibre axis. These two orientations result from a competition between the orientational tendencies of the smectic layers and of the polymer chains.

1. Introduction

Many liquid-crystalline polymers have been synthesized and investigated in recent years. These polymers include polymers with mesogenic groups in the main chain or as part of the side groups [1, 2], or polymers with more complex structures [3]. X-ray investigations of liquid-crystalline polymers are important for an accurate identification of the liquid-crystalline phases [4, 5] and for the elucidation of the molecular packing. In addition it is easy to determine the molecular orientation in different samples and thus to learn something about the orientation of these materials under mechanical deformation. Since a bidirectional orientation has been reported [6] for fibres of one combined liquid-crystalline polymer [6, 7] (polymers with the mesogenic groups in the main chain as well as in the side groups), it was especially interesting to investigate polymers of this type in more detail and to compare them with conventional liquid-crystalline main chain polymers.

Here we describe the results of X-ray measurements on linear (non-cross-linked) and cross-linked liquid-crystalline main chain (2 and 3) and combined polymers (4-6), the synthesis of which is described elsewhere [8, 9]. The molecular structure and the characterization of these polymers is presented in tables 1 and 2. The results of the

Table 1. Linear liquid-crystalline polymers **2** and **3** with the mesogenic groups in the main chain and the corresponding cross-linked polymers **2'** and **3'**.

Polymer	Molecular weight†	Amount of cross-linking agent 1 used in mole %	Phase transitions‡ in °C
2 (sample 1)	14000	—	K ₁ 87 S _B 104 S _A 121 I
2' (sample 1)	—	18	K ₁ 76 S _B 98 S _A 115 I
2 (sample 2)	9000	—	K ₁ 91 S _B 108 S _A 125 I
2' (sample 2)	—	7	K ₁ 74 S _B 109 S _A 120 I
3	20000	—	K ₁ 34 S _A 132 I
3'	—	10	K ₁ 31 S _A 128 I

† GPC in chloroform calibrated against polystyrene standards.

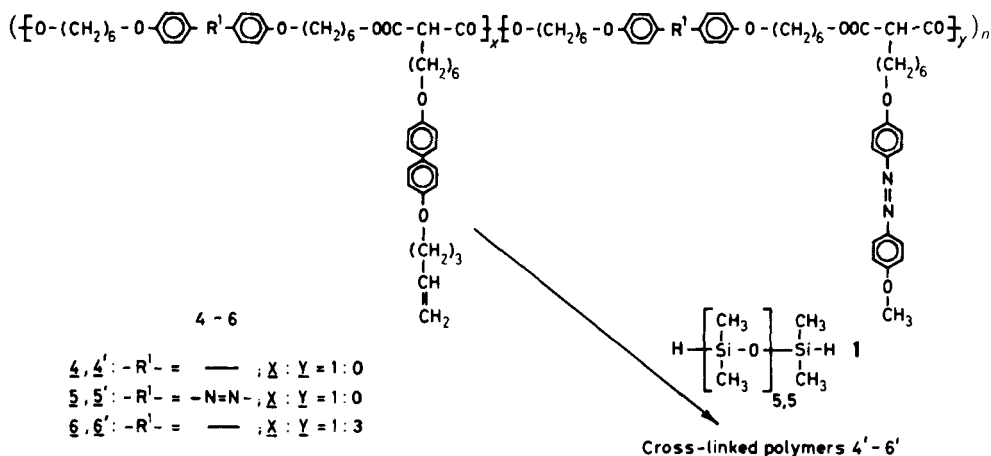
‡ K₁, crystalline or highly ordered smectic phase (mesogenic groups perpendicular to the layers); S_B, smectic B; S_A, smectic A; I, isotropic.

X-ray measurements are already taken into account in the phase assignments. Oriented specimens were investigated with a flat-plate camera. The measurements were performed for the cross-linked polymers at different temperatures in the liquid-crystalline phases; for the linear polymers they were only possible at room temperature. In addition temperature dependent X-ray measurements were performed with unoriented samples of the linear polymers.

2. Experimental

The thermotropic behaviour of the linear (non-cross-linked) and cross-linked polymers was studied with a Perkin-Elmer DSC-2C differential scanning calorimeter (peak maxima) and by polarizing microscopy (Leitz, optical microscope POL BK-II, in combination with a Mettler FB2 hotstage). The X-ray experiments were performed using Ni filtered CuK_α radiation. The temperature dependent measurements for the oriented specimen were done in a sample holder (which could be heated with thermostatted air) that allowed the sample to be stretched and rotated (around the fibre axis) during the measurements, in combination with a flat-plate camera. The temperature dependent measurements of unoriented samples were performed with a Siemens D 500 X-ray powder diffractometer in combination with a heatable sample holder, which was evacuated during the measurements. The determination of the small angle reflections could be made more accurately with the diffractometer than with the flat-plate camera. Fibres of the non-cross-linked polymers **2–6** were drawn from the melt. Ribbon-like samples of the cross-linked polymers **2'–6'** were fastened to the heatable sample holder and investigated in the different liquid-crystalline phases under stress. Alternatively the initially unoriented samples were fastened to a non-heatable stretching device. They were then heated to the isotropic phase (determined

Table 2. Linear combined liquid-crystalline polymers 4-6 (mesogenic groups in the main chain and as side groups) and the corresponding cross-linked polymers 4'-6'.



Polymer	Molecular weight†	Amount of cross-linking agent 1 used in mole %	Phase transitions‡ in °C
4	> 80000	—	K ₁ 162 S _A 170 I
4'	—	5	K ₁ 150 S _A 158 I
5	> 80000	—	K ₂ 124 S ₁ 150 S _A 154 I
5'	—	10	K ₂ 119 S ₁ 148 I§
6	80000	—	K ₂ 126 S ₁ 143 S _A 154 I
6'	—	10	K ₂ 122 S ₁ 136 S _A 149 I

† GPC in chloroform calibrated against polystyrene standards.

‡ K₁, K₂, crystalline or highly ordered smectic phase (K₁, orthogonal; K₂, tilted); S_A, smectic A; S₁, probably smectic C; I, isotropic.

§ The transition S₁ to the isotropic phase is broadened and not resolved.

optically) with a hot air blower and stretched. They were maintained in the isotropic phase for some minutes, cooled slowly to room temperature (corresponding to a crystalline or highly ordered smectic phase) and were investigated. Both procedures resulted in the same fibre pattern at room temperature. The length of the molecules was determined with the help of Dreiding models measured in the most extended state.

3. Results

3.1. Phase assignment

The temperature dependent X-ray measurements give the results presented in figure 1. The results for polymer 2 are already given in [8]. Fibre diagrams of the linear (non-cross-linked) and cross-linked polymers at room temperature are shown in figures 2, 3, 5 and 7.

At room temperature all of the polymers are in a crystalline or highly ordered smectic phase. The X-ray diagrams are dominated by a sharp, small angle reflection ($2\theta = 2-4^\circ$) and two sharp, wide angle reflections ($2\theta \approx 20^\circ$ or 23°) corresponding to lattice spacings of $3.8 \pm 0.1 \text{ \AA}$ and $4.4 \pm 0.1 \text{ \AA}$. In addition all polymers show a weak and broad wide angle reflection corresponding to a Bragg spacing of about

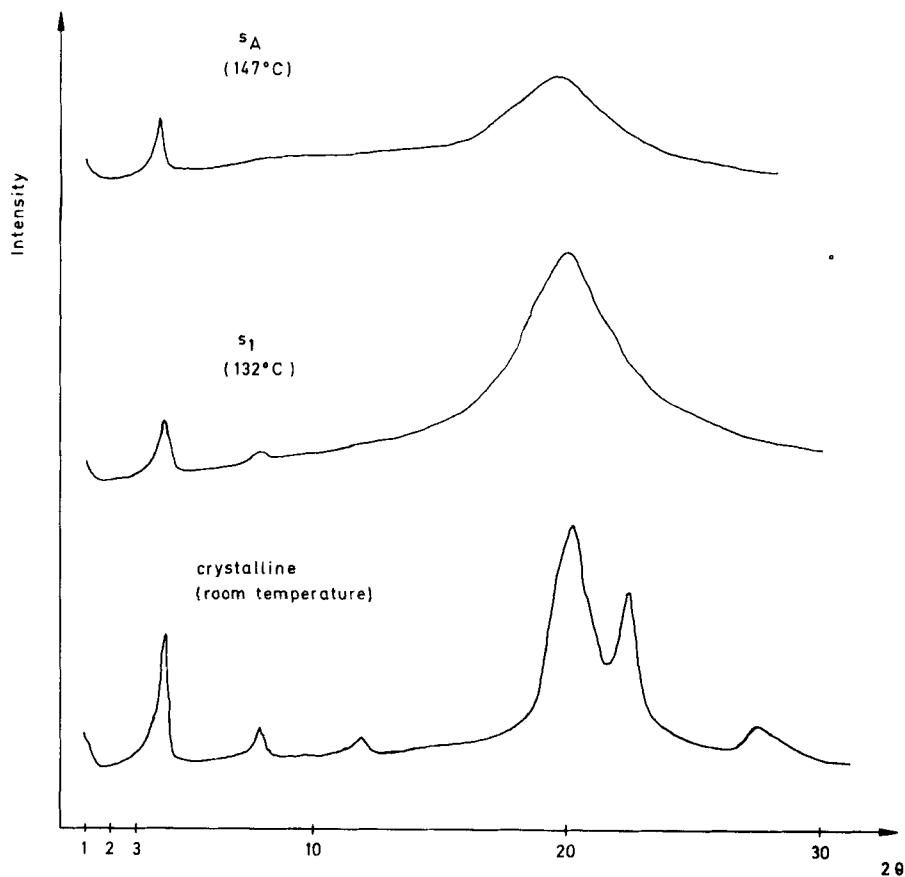


Figure 1. Temperature dependent X-ray measurements for polymer **6**, the intensity is in arbitrary units.

3.2 Å. The layer spacings calculated from the small angle reflections are summarized in tables 3 and 4. In comparison with low molar mass liquid crystals the orientation is at least better than in hexatic smectic **B** phases (pseudo-hexagonal packing, with only one sharp, wide angle reflection [4, 5]). Polymer **2** (see [8]) shows a series of additional reflections at intermediate angles; polymer **3** shows one strong reflection at $2\theta = 10^\circ$; the polymers **4–6** show no additional reflections.

From the fibre pattern of the linear and cross-linked polymers at room temperature (cf. figure 2, 3, 5 and 7) it can be seen that the reflections corresponding to 4.4 Å are either orthogonal (figures 2, 3 and 7) or tilted (figure 5) with respect to the small angle layer reflection. This behaviour is not affected by the cross-linking. Thus the long axes of the mesogenic groups are either parallel (polymers **2–4**) or tilted with respect to the layer normal (polymers **5** and **6**). The tilt angle as evaluated from the fibre diagrams is $27^\circ \pm 2^\circ$ for both polymers independent of the cross-linking. Therefore a transition to a phase with an orthogonal orientation of the mesogenic groups should lead to an increase of the layer spacing of about 3 Å. The broadness of the reflections and the accurate layer spacings depend on the history of the sample and can be changed by annealing. For some polymers the wide angle reflections become reversibly sharper with increasing temperature.

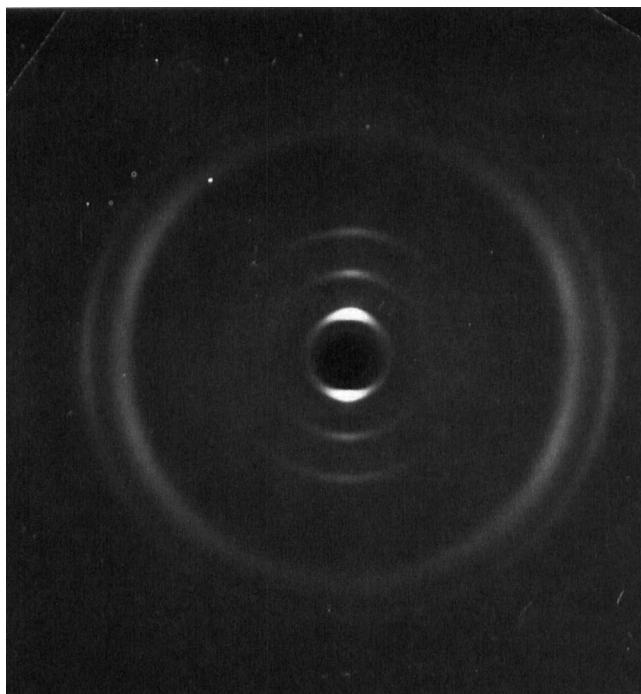


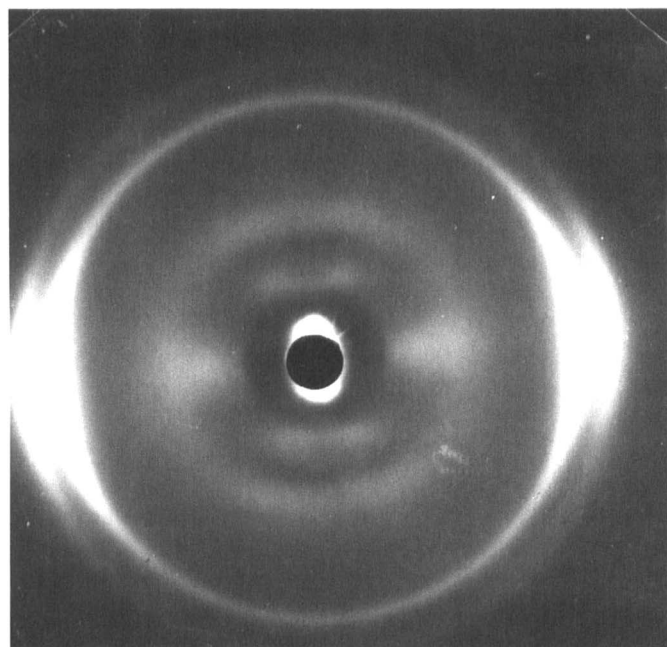
Figure 2. Fibre pattern of the non-cross-linked polymer 4 at room temperature; distance from the fibre to the film was 83 mm and the fibre axis was vertical.

Table 3. Layer spacing derived from the small angle reflection of the linear and cross-linked polymers 2 and 3 (see table 1).

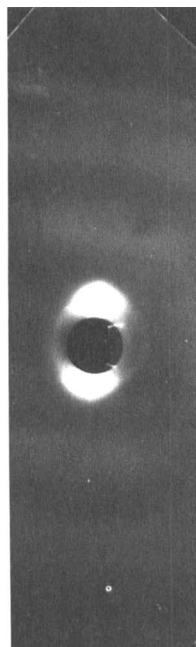
Polymer	Temperature/°C	Phase	Layer spacing/Å
2 (sample 1 + 2)	RT	K ₁	28–30
	95	S _B	30.5 ± 0.5
	115	S _A	30.5 ± 0.5
2' (sample 1)	RT	K ₁	44.0 ± 1.0
2' (sample 2)	RT	K ₁	33.5 ± 1.0
	90	S _B	35.0 ± 1.0
	116	S _A	34.5 ± 1.0
3	RT	K ₁	32.5 ± 0.5
	60	S _A	32.5 ± 0.5
3'	RT	K ₁	36.5 ± 1.0
	60	S _A	36.0 ± 1.0

RT ≡ room temperature.

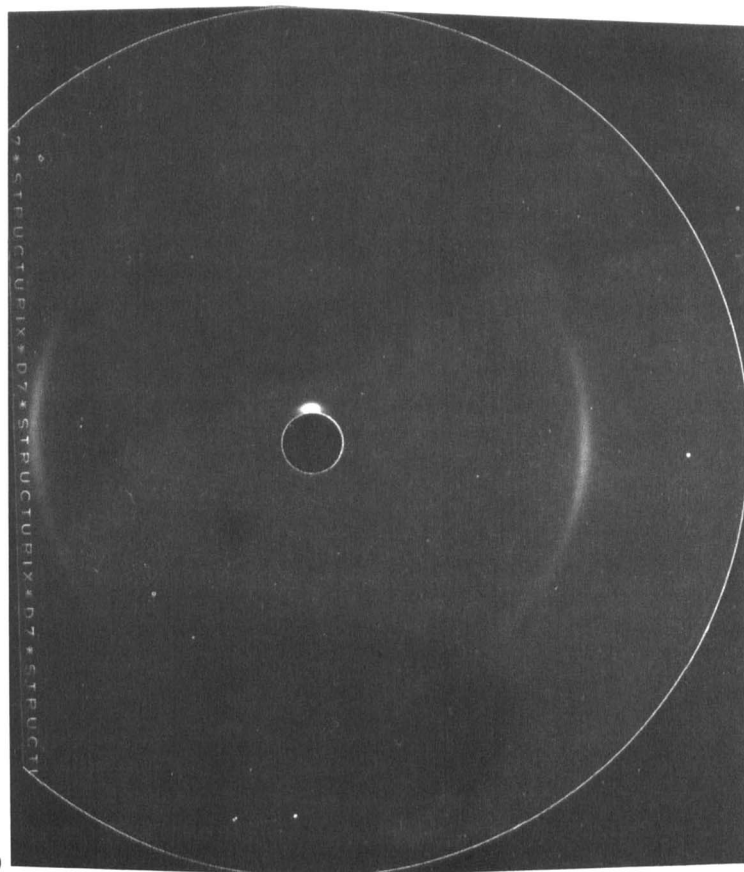
The X-ray diagram of the unoriented phase designated as smectic B (polymer 2, see [8]) consists of one sharp, small angle reflection (see table 3) and only one sharp, wide angle reflection corresponding to 4.4 ± 0.1 Å. The layer spacing and the lateral dimension remain unchanged compared to the crystalline state at room temperature. The preservation of the orthogonal arrangement of the long axes of the mesogenic groups to the layer structure can be seen clearly from the fibre pattern of polymer 2' (see figure 3). In analogy to low molar mass liquid crystals [4, 5], this phase should be



(a)



(b)



(c)

Figure 3. Fibre pattern of the cross-linked polymer **2'** (sample 2), strain ratio 40 per cent, fibre axis vertical; crystalline phase at room temperature, fibre-film distance (a) 94 mm, (b) 130 mm; (c) smectic B phase at 90°C, fibre-film distance 100 mm.

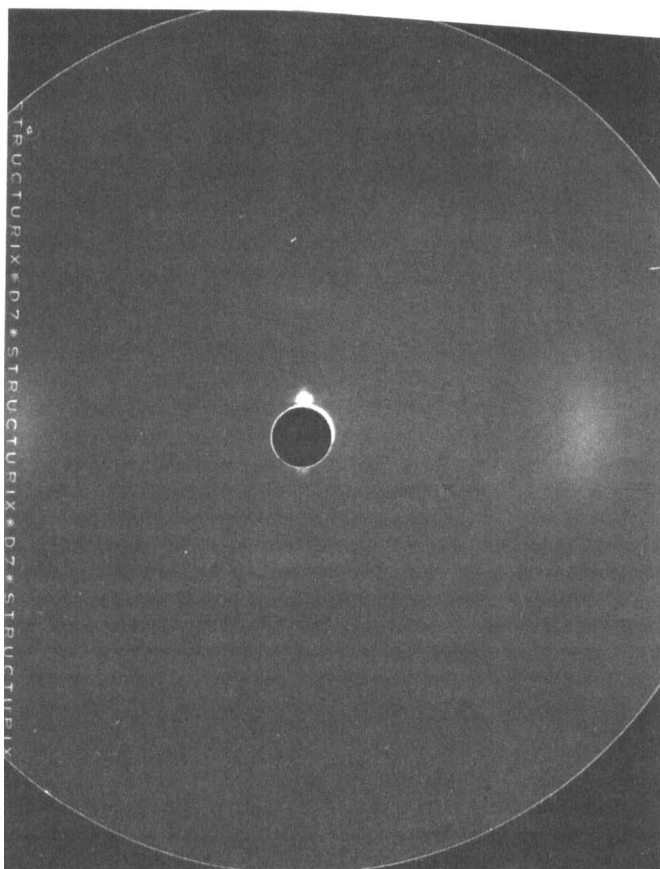
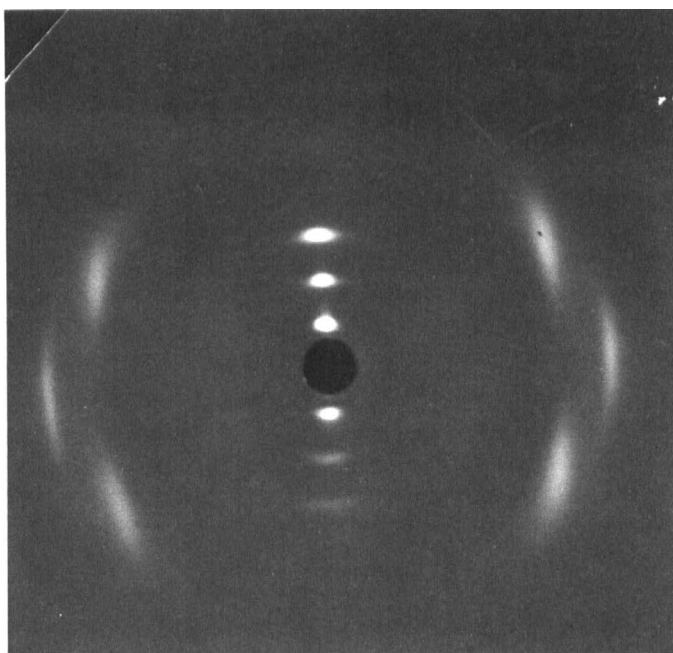


Figure 4. Fibre pattern of the cross-linked polymer 3' at 59°C in the smectic A phase; fibre-film distance 100 mm, fibre axis vertical.

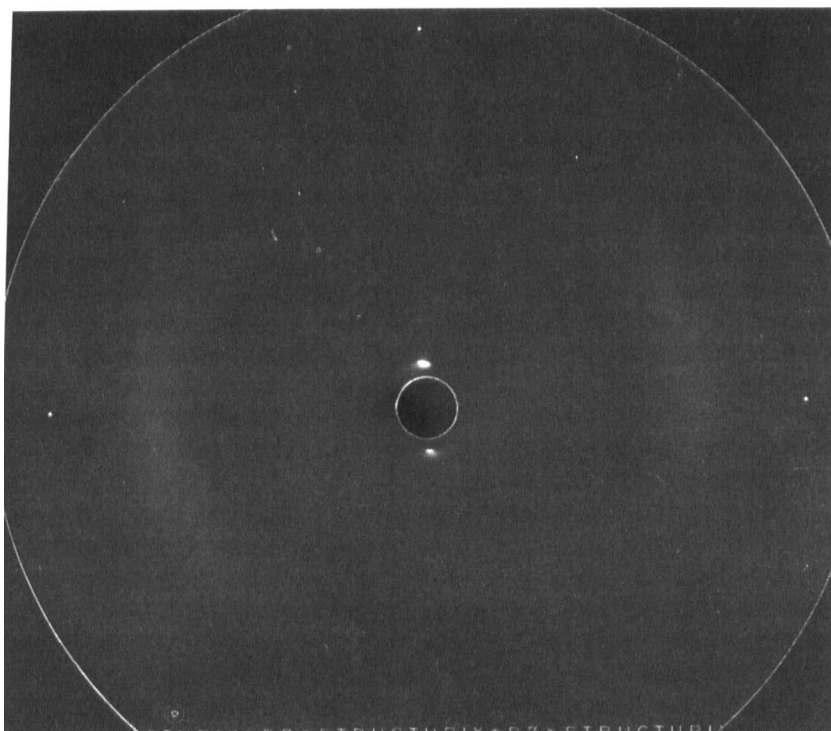
Table 4. Layer spacing derived from the small angle reflection of the linear and cross-linked polymers 4-6 (see table 2).

Polymer	Temperature/°C	Phase	Layer spacing/Å
4	RT	K ₁	23.0 ± 0.5
	168	S _A	24.0 ± 0.5
4'	RT	K ₁	24.0 ± 1.0
5	RT	K ₂	22-23
	140	S ₁	23.5 ± 0.5
	153	S _A	24.0 ± 0.5
5'	RT	K ₂	24.0 ± 1.0
	132	S ₁	25.0 ± 1.0
	141	S ₁	25.5 ± 1.0
6	RT	K ₂	21.5 ± 0.5
	132	S ₁	22.5 ± 0.5
	147	S _A	23.5 ± 0.5
6'	RT	K ₂	23.0 ± 1.0
	128	S ₁	24.5 ± 1.0
	142	S _A	25.5 ± 1.0

RT ≡ room temperature.



(a)



(b)

Figure 5. Fibre pattern of the cross-linked polymer **5'**, fibre axis vertical: (a) crystalline phase at room temperature, fibre-film distance 94 mm; (b) S_1 phase at 141°C, fibre-film distance 100 mm.

a smectic B phase, with a hexagonal packing of the long axes of the mesogenic groups orthogonal to the smectic layers. In contrast to the crystalline phase, no influence of the history of the sample on the X-ray diagram was observed.

The X-ray diagrams of the unoriented phases designated as smectic A consist of one sharp, small angle reflection and one broadened, wide angle reflection corresponding to a Bragg spacing of 4.5 \AA (see figure 1). For the polymers 2–4, the layer spacing is essentially unchanged compared to the layer spacing at room temperature (see tables 3 and 4), where an orthogonal arrangement of mesogenic groups and layer structure is observed. The orthogonal orientation of the smectic layers and the mesogenic groups can be seen clearly from the fibre pattern (cf. figure 4).

For the polymers 5 and 6 the increase of the layer spacing at the transition from the tilted crystalline to the first smectic phase (S_1) is smaller than expected (see table 4) for a transition to an orthogonal smectic phase (S_A). In addition there is a second smectic phase (S_A) at higher temperatures and both phases show the characteristic X-ray pattern of smectic A or C phases for unoriented samples (see figure 1). The phase transition between these two smectic phases can be seen clearly by polarizing microscopy (fan shaped texture S_A ; broken fan shaped texture, S_1) and for polymer 6 by D.S.C. measurements (see figure 6). For polymer 5 the transition between both smectic phases is very close to the clearing temperature and cannot always be well-resolved by D.S.C. measurements. Therefore the existence of a smectic C phase at lower temperatures and of a smectic A phase at higher temperatures seems reasonable [8]. The fibre pattern of oriented samples of the cross-linked polymers 5' and 6' show however for all smectic phases an orthogonal orientation of the small angle reflections

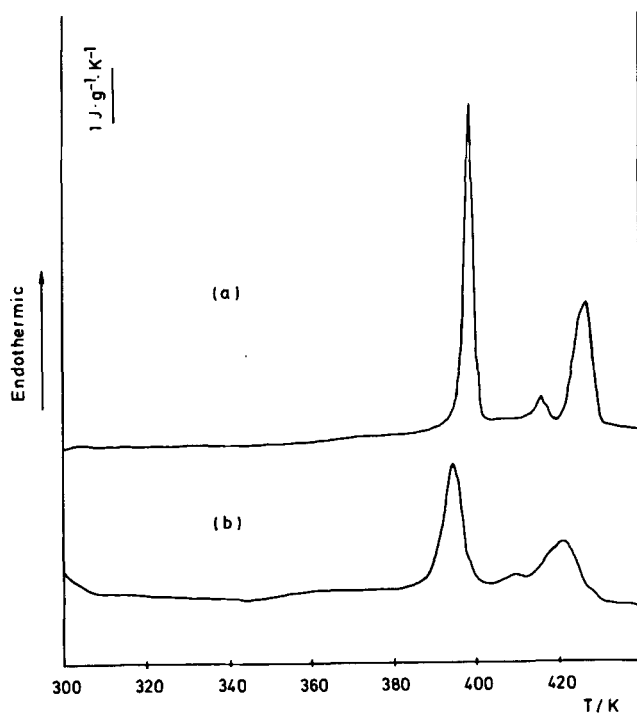


Figure 6. D.S.C. measurements of (a) the non-cross-linked polymer 6 and (b) the corresponding cross-linked polymer 6' (see table 2); heating rate 10 K/min.

(layer structure) and the broadened wide angle reflection (lateral packing, see figure 5). This cannot exclude a smectic C phase [14] at lower temperatures (S_1), but it also points towards two phases of the smectic A type, in this case their difference is not clear. Diffractometer measurements (see figure 1) show only a small shift of the small angle reflection for about 1 \AA (see table 4) and a broadening of the wide angle reflection at the transition from the S_1 to the S_A phase.

3.2. Effect of cross-linking

A comparison of the fibre pattern of linear (non-cross-linked) and cross-linked polymers shows that the cross-linking has no influence on the type of pattern, but that it leads to an increase of the layer spacing for the liquid-crystalline main chain polymers **2** and **3**. For amounts of the cross-linking agent **1** from 7 to 10 per cent (polymer **2'** (sample 2) and polymer **3'**) the layer thickness is increased by $3\text{--}4 \text{ \AA}$, while for higher amounts of the cross-linking agent (polymer **2'** (sample 1)) it is increased by 14 \AA (see table 3). For the combined polymers **4–6** the cross-linking has no significant influence on the layer spacing. The behaviour of the combined polymers is therefore similar to that of liquid-crystalline side group polymers [10].

3.3. Comparison of main chain and combined polymers

The fibre pattern of the combined polymers (cf. figures 2 and 5) are analogous to the fibre pattern of the main chain polymers (cf. figures 3, 4 and 7). They can be understood in analogy to low molar mass liquid crystals [4, 5]. No bidirectional

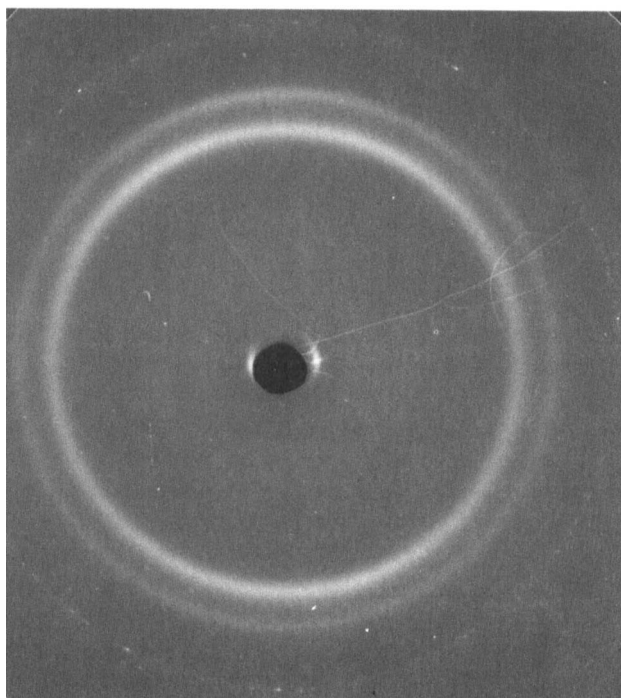


Figure 7. Fibre pattern of the non-cross-linked polymer **2** at room temperature; fibre–film distance 83 mm ; fibre axis vertical.

orientation [6] is observed. However two different orientations either with the smectic layers (figure 7) or the long axes of the mesogenic groups (figures 2–5) parallel to the fibre axis can be found for main chain and combined polymers, depending on the molecular weight or the strain ratio.

3.4. Strain-induced orientation

Stretched fibres of the linear main chain polymers **2** and **3** and of the linear combined polymer **6** show a fibre pattern with the small angle reflection at the equator perpendicular to the drawing direction. Thus, the smectic layers are oriented in the direction of strain (see figure 10(a)). In contrast to these polymers with a low molecular weight, highly stretched fibres of the linear polymers **4** and **5** and of all cross-linked polymers (**2'**–**6'**) show a fibre pattern with the small angle reflections at the meridian parallel to the drawing direction. Thus, in this case the long axis of the mesogenic groups and thereby the polymer main chains are oriented parallel to the fibre axis (see figure 10(b)). For the cross-linked polymer **2'** (sample 1), a transition between these two orientations was observed, depending on the strain ratio. For the small strain ratio of 8 per cent a weak orientation resulted with the maxima of the small angle reflections close to the equator. This corresponds to fibres of the linear polymer **2**. Thus at this stage the smectic layers are oriented with a small preference parallel to the direction of strain. For strain ratios of more than 10 per cent, however, the small angle reflections are observed at the meridian (see figure 3). Now of course the polymer chains are oriented preferentially parallel to the direction of the strain.

4. Discussion

4.1. Phase assignment

The assignment of the high temperature smectic phases was possible in most cases by evaluation of the temperature dependent X-ray measurements and polarizing microscopy. For the polymers **5** and **6** with two different disordered smectic phases the phase assignment is complicated. Although different types of smectic A phases with monolayers, interdigitated structures or double layers have been reported [4], the nearly unchanged layer thickness ($\approx 1 \text{ \AA}$) makes the understanding of a phase transition difficult if two smectic A phases are considered. In addition the X-ray pattern of a multidomain sample of a smectic C phase, in which the layers are oriented but the tilt direction takes all possible orientations (see figure 8), can hardly be distinguished from the X-ray pattern of a smectic A phase [14]. The only difference between the fibre pattern of both phases is, in this case, the broader angular distribution of the wide angle reflection in the smectic C phase, corresponding to a smectic A phase of a reduced order parameter. Combining all the results, we believe that it is most reasonable that the S_1 phase has a tilted structure and is of the smectic C type.

For polymer **2** a crystalline phase is reasonable at room temperature because of the multitude of sharp reflections (see [8]). For polymers **3**–**6** highly ordered smectic phases should also be considered. From the fibre pattern at room temperature it is clear that the layer structure is very well established, whilst the molecular packing in the layers is less well defined. This can be described according to Hosemann and Bagchi [11] as a paracrystal with a highly anisotropic degree of disorder. According to low molar mass liquid crystal terminology smectic E (polymers **3** and **4**) or smectic H phases (polymers **5** and **6**) also seem possible [4, 5].

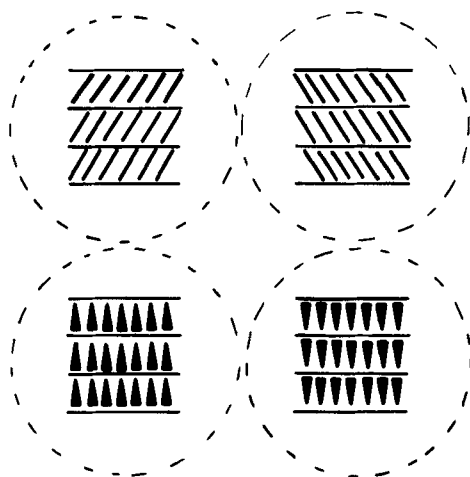


Figure 8. Multidomain sample of a smectic C phase, in which the layers are oriented, but the tilt direction takes all possible orientations; \blacktriangle or \blacktriangledown : mesogenic groups are tilted with respect to the drawing plane.

4.2. Comparison of the X-ray spacings with the molar geometry

The spacing of the smectic layers of the linear liquid-crystalline main chain polymers **2** and **3** (see table 3) agrees well with the length of the repeating unit along the polymer chain, as determined with the help of Dreiding models. In this way a length of $31 \pm 1 \text{ \AA}$ was determined for polymer **2**, and a length of $33 \pm 1 \text{ \AA}$ for polymer **3**. Thus, the smectic layers are formed by monolayers of the repeating units. The reason for the increase of the layer thickness after cross-linking is not yet clear. In analogy to low molar mass liquid crystals, this might mean a transition from a monolayer to an interdigitated structure [4].

The length of the mesogenic side groups (from the polymer chain to the very end) is less than the length of the repeating unit in the main chain for the liquid-crystalline combined polymers **4–6**. The length of the side groups in polymers **4** and **5** is $24 \pm 1 \text{ \AA}$, as determined with Dreiding models. The length of the side group of the comonomer unit of polymer **6** is $21 \pm 1 \text{ \AA}$. The experimentally observed layer spacing (see table 4) is very close to the length of the side groups. Nevertheless, there is no evidence that the smectic layers in the combined polymers are more poorly defined than in the main chain polymers. This result and the complete analogy between the fibre pattern of the liquid-crystalline main chain and combine polymers imply an arrangement of the mesogenic groups as illustrated in figure 9. This structure with the mesogenic groups in the main chain and in the side groups parallel to each other has already been favoured because of texture observations and the mesophase broadening [6, 7]. However a locally bidirectional structure as proposed in [6], in which the mesogenic groups in the side groups and in the main chain are oriented perpendicular to each other and form two orthogonal sets of layers, should lead to two different layer thicknesses and should retain the bidirectional fibre pattern [6] for the oriented samples under investigation. It may therefore, be ruled out. The bidirectional fibre pattern observed for some combined polymers must therefore result from a macroscopic superposition of two different orientations.

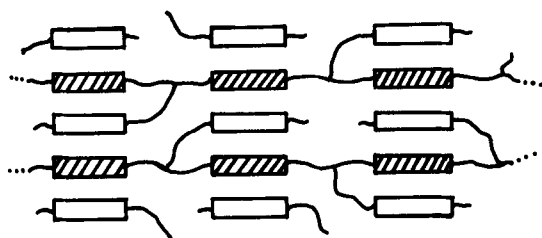


Figure 9. Proposed arrangement of the mesogenic groups in the main chain and in the side groups for combined liquid-crystalline polymers.

4.3. Strain-induced orientation

The occurrence of two different orientations either with the polymer chains or the layer structure parallel to the fibre axis is well-known for partially crystalline polymers [12] and for liquid-crystalline main chain polymers with smectic phases [13]. For the polymers under investigation it results from the competition of the tendency of the smectic layers to slide over each other and of the tendency of the polymer chains to orient in the direction of drawing (see figure 10). For polymers of a low molecular weight entanglements which favour an orientation via the polymer chains are not yet very effective. Consequently, the orientation is dominated by the sliding of the smectic layers in analogy to low molar mass liquid crystals, although this orientation is unfavourable for the polymer chains (see figures 7 and 10(a)). For polymers of a higher molecular weight, and consequently for cross-linked polymers, the orientation is dominated by the polymer chains for high strain ratios (see figure 10(b)). For cross-linked polymers at low strain ratios the deformation of the chains may still be small. Then the tendency of the smectic layers to orient in a strain field will dominate. In an intermediate range of molecular weight and strain, both orientations may

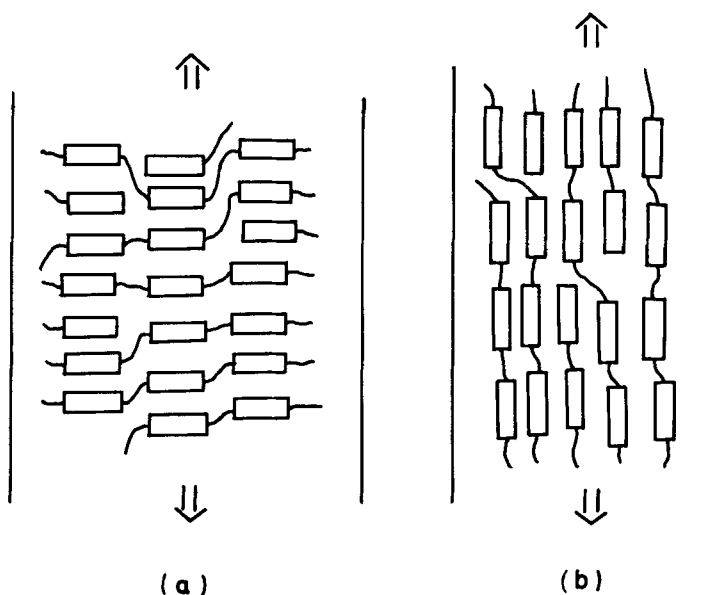


Figure 10. Possible orientation with (a) the smectic layers or (b) the polymer chain parallel to the fibre axis (direction of strain).

appear simultaneously [6]. This is probably due to differences between the surface and the bulk of the sample during the preparation of the fibre.

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