Liquid Crystals
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713926090

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Online publication date: 16 August 2010

To cite this Article Meyer, C. , Logbo, H. , Briouel, B. , Picot, J. -C. , Nastishin, Yu.A. and Kleman, M.(2010) 'Double helical defects in smectic A and smectic A* phases', Liquid Crystals, 37: 8, 1047 — 1057
To link to this Article: DOI: 10.1080/02678291003782846
URL: http://dx.doi.org/10.1080/02678291003782846

Liquid Crystals,
Vol. 37, No. 8, August 2010, 1047–1057

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Double helical defects in smectic A and smectic A* phases

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(*Received 5 February 2010; final version received 16 March 2010*)

Double helical (DH and DH*) domains, defects of the smectic A (SmA) phase, have for the first time been studied in the smectic A* (SmA*) phase, composed of chiral molecules. Both left- and right-handed DH*s are found in SmA*s, but not in equal numbers. The majority of the DH*s in the two left-handed cholesterics studied are left-handed, whereas in the SmA phase of non-chiral molecules the DHs are left- and right-handed in equal numbers. DHs and DH*s, which are topologically screw dislocations of giant Burgers vectors, appear to be significantly distorted with respect to a geometrically ideal model. It has been shown that imperfections in the DH*s and DHs are covered by a combination of three types of elementary distortions. The relation between the pitch in DH* and DH and temperature is discussed.

Keywords: chiral smectic A; defects; double helices; screw dislocations

1. Introduction

The point group of a non-chiral smectic A (SmA) is ∞/mm (or $D_{\infty h}$ in other notation) [1]. Although there is no difference between the structure of the director field and the smectic layering in the ground state of a SmA*, comprising chiral molecules, the symmetry group is $\infty 2$ (or D_{∞}) [1]. It has been recommended by Baron *et al.* that the smectic A phase in chiral materials should be identified by a star (SmA*) to distinguish it from the non-chiral smectic phase (SmA) [2].

The difference in symmetry implies a corresponding difference in physical properties, for example the electroclinic effect is possible in SmA* but forbidden in a SmA [3]. Another example lies in structural singularities. SmA phases often exhibit well-known macroscopic defects in the shape of the double helix (DH) [4]. At first glance the double helices encountered in SmAs and in SmA*s may appear similar, but closer examination shows that most DH*s have characteristics which differentiate them from DHs.

The present paper summarises the properties of the double helical domains, placing them within the perspective of the so-called isometric defects of SmAs. Observations are offered to describe the specificities of DH* and to stress the importance of chiral properties *vs* isometry. The interplay between chirality and isometry, and how this affects defects in SmA*s, will be developed in a future paper.

Defects in smectics are described as isometric if they preserve the parallelism and equidistance of the layers [5]. The construction of an isometric domain obeys the rule that, starting from a parent layer of any shape, all the layers parallel and equidistant to this layer constitute an isometric solution. This conserves the ground state distance d_0 between the layers, which generically cannot extend to infinity (unless of course the layers are planar), since this is limited by the two focal sheets where the layers have infinite curvature, and thereby infinite energy density. Thus isometric geometries are limited to domains of finite extent.

In certain respects DHs and DH*s belong to such a family, but the pure isometric case (apart from the trivial ground state case) is that of focal conic domains (FCD), which may be briefly summarised as follows. FCDs consist of a pair of singular lines (two cofocal conics, an ellipse and a hyperbola) about which the smectic layers fold into Dupin cyclides. The ellipse and the hyperbola are visible under the light microscope; in special cases they degenerate into a circle and a straight line. They were first observed and described by Friedel and Grandjean [6]. Recent reviews of the topic have been provided by Kleman and Lavrentovich [7, 8].

The evident perfection of these objects gives the impression of geometric ideality and as a consequence creates the illusion that they are fully understood. Imperfections in the ideality of FCDs have been recently investigated [9–11]; some of these are macroscopic, but most are microscopic, described as "kinks".

Ideal DHs (and DH*s) are cylindrical isometric domains; they comprise a stack of layers parallel to ruled helicoids and are limited to the cylinder (Figure 1); this is described in greater detail later. But

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ISSN 0267-8292 print/ISSN 1366-5855 online

DOI: 10.1080/02678291003782846
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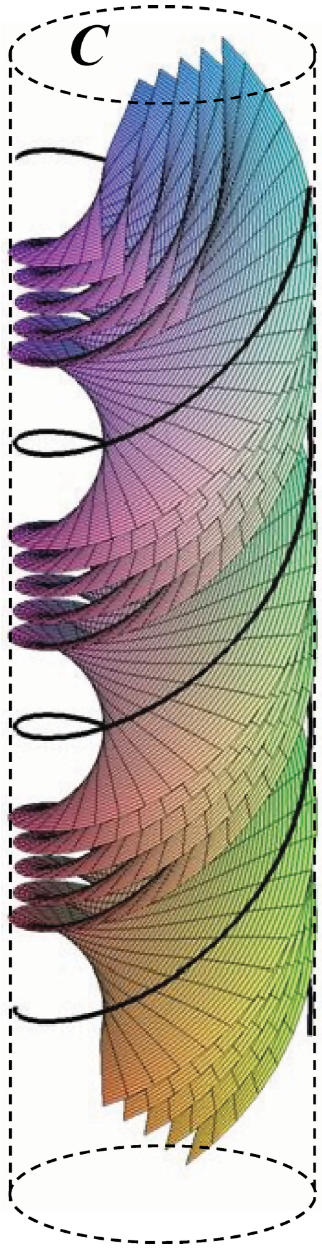

Figure 1. Parallel equidistant layers fill the cylinder C; the only defects in this stacking are the two helical disclinations (thick solid lines) of strength ½ on the cylinder surface. The gaps between the represented layers are purposely left unfilled in order to show the shape of the layers; it should be understood that non-intersecting equidistant layers, parallel to those shown, can be introduced inside these gaps.

DHs and DH*s are practically always imperfect on the macroscopic scale, and we shall see that the imperfections differ between the SmA and SmA* phases.

It should be noted that the integrated energy of an isometric domain is certainly reduced if (singular) sheets degenerate to (singular) lines. This is the situation encountered with FCDs, whereas in the case of DH domains the singular focal sheets have surfaces which exhibit a cusp along two helical lines (thick lines in Figure 2(a) and (b)) located on the surface of the cylinder *C* (depicted by thin dashed lines), and which make multiple intersections outside *C* (Figure 2(c)). These singular focal sheets cannot degenerate into lines. However, the energy penalty for these surface singularities can be reduced if the DH domain does not extend beyond *C*, thereby making the singular focal sheets virtual. In this case the only singularities physically present are two helical lines on *C* – the cusp lines mentioned above[1].

A FCD can be smoothly embedded into a matrix of parallel smectic layers (representing the smectic ground state) without any additional defects inside it; thus most observed FCDs are reasonably ideal inside the domain and on its boundary. By smooth embedding is meant that at the boundary of the FCD the layer curvatures are continuous, therefore strictly vanishing in the present case. But this is not so for a DH. Deformations of relaxation are therefore expected, and these are the origin of distortions within the cylinder *C*, distortions of the cylinder itself, and outside it, shared in different ways according to the type of material; the B7 phase alluded to in Footnote 1 is a typical example. To summarise, DHs are rarely ideally isometric, in the sense that the layer parallelism is rarely ideally satisfied inside *C*.

Outside *C* the smectic layers have to adopt a different geometry, which intuitively should correspond to the ground smectic state, i.e. the DH should be embedded into a matrix of parallel (or, more correctly, quasi-parallel) smectic layers. Altogether these paired helices dressed with a family of quasi-parallel layers outside the central cylinder constitute topologically a mode of splitting a screw dislocation of a giant Burgers vector b into two disclination lines of strength $k = ½$**,** the cusp lines above, about which the layers are rotating**.** More precisely, the Burgers vector is equal to the pitch of an ideal DH from which the actual DH can be constructed through the relaxation deformations just mentioned:

$$b = p, \tag{1}$$

where p is the pitch.

Opposite pitches correspond to screw dislocations of opposite Burgers vectors. Details are given in Reference [12].

The objectives of the present study were threefold:

1. To determine experimentally whether the chirality of the material influenced the ratio between the numbers of right- and left-handed double helices in a SmA*. In the course of this study DH*s have been compared with DHs.

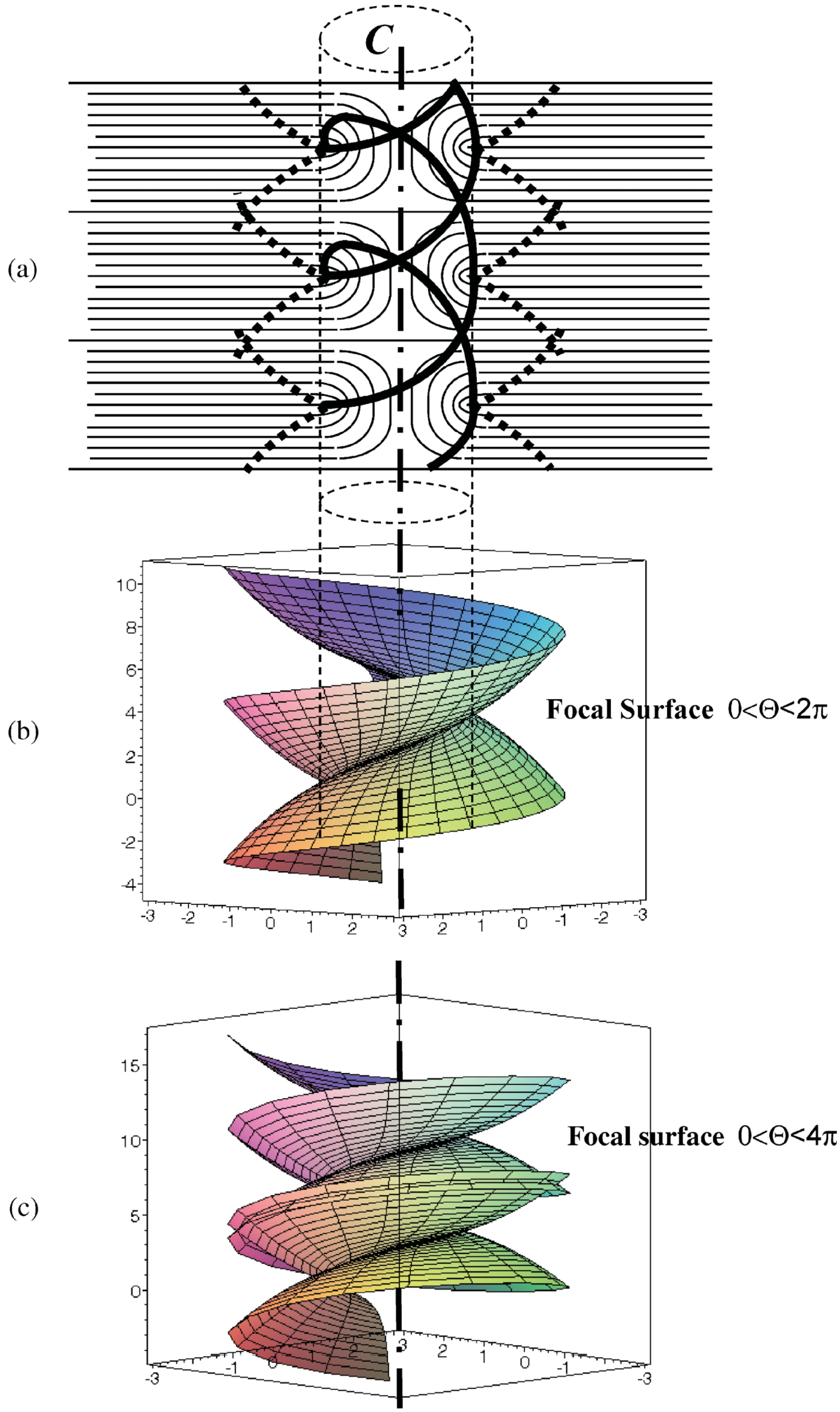

Figure 2. Singularities of the DH domain: (a) sketch of the DH domain bounded by the cylinder C (thin dashed lines) and embedded into a matrix of quasi-parallel smectic layers (thin lines); the thick dashed lines show the intersection of the focal surfaces with the plane of the figure; (b) a segment of the focal sheets; (c) an extended image of the focal sheets (colour version online).

2. To investigate whether the pitch of the double helix was sensitive to temperature. It appears that the behaviour of DHs and DH*s varies with temperature.
3. To classify imperfections in the double helical domains experimentally observed in the SmA and SmA* phases in the light of points 1 and 2.

2. Experimental

Two cholesterol derivatives which exhibit a SmA* phase on cooling have been investigated: cholesteryl nonanoate (CN) and cholesteryl tetradecanoate (CT). The observed DH*s have been compared to the DHs in SmAs of the non-chiral materials 4-*n*-octyl-4′-cyanobiphenyl (8CB) and 4-*n*-nonyl-4′-cyanobiphenyl (9CB). On cooling from the cholesteric phase CN has been reported to display anomalous properties as it approaches the transition to the SmA*, and due to these anomalies it is claimed to be an analogue of a superconductor of Type II [13–17]; in particular it displays a twist grain boundary type A (TGBA) phase between the SmA* and the N* phases [14, 15]. CT does not show such anomalies and is a typical analogue of a Type I superconductor. Observations on these two materials have been compared to establish the differences in the formation of double helices and their properties.

The samples were observed under a polarising optical microscope in transmission mode. The temperature of the sample was controlled using the Instec Hot Stage (HS–1), to within an accuracy of 0.01°C. If the sample was cooled from the cholesteric phase with planar texture, in which the helical axis of the cholesteric structure is perpendicular to the substrates, it was seen that the double helix in the smectic phase appeared to be oriented with its helical axis parallel to the substrates. If the molecules at the substrates have homeotropic orientation, then after cooling, the smectic layers lie parallel to the substrates and the double helices become oriented with their helical axes perpendicular to the substrates.

It is more convenient to study the double helix when it is lying on its side with the helical axis parallel to the surfaces, and we therefore need planar orientation of the molecules at the substrates. Typical nematic alignment techniques for planar directional orientation using rubbed polymers do not work for the cholesteric phase of these materials. The easiest way to produce a planar texture is to shift the upper substrate. A cell has been constructed which allows the upper substrate to be shifted using a step motor without opening the hot stage, keeping the cell thermostable and suitable for microscope observation. The thickness of the cell was fixed using 75 μm thick Mylar strips.

3. Results and discussion

3.1 DH helicity: left versus right*

Both right- and left-handed double helices (Figure 3 (a) and (b), respectively) were found in CN and CT samples, but not in equal number. In the case of CN (a left-handed material) we found that left-handed DH*s were much more numerous – among 100 DH*s examined, 80 were left-handed and only 20 were right-handed. On the other hand, double helices of both handedness appeared in approximately equal numbers in non-chiral smectic materials. (To allot a sign to the handedness of a DH the so-called right-hand grip rule [18], employed in the theory of electromagnetism, has been adopted.)

It has been concluded in the present study that for the SmA* phase the ratio of left- and right-handed helices is the result of the presence of the residual twist of the director field remaining from the cholesteric N* phase. In the N* phase the director field is twisted above the transition. Approaching the phase

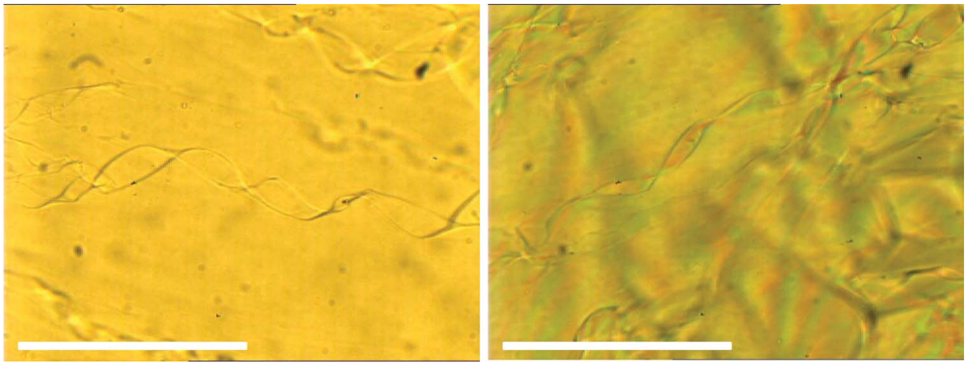

Figure 3. Left and right DH*s in the SmA* phase of CN; light microscopy, parallel polarisers. The white bar corresponds to 100 μm (colour version online).

transition, and during the transition itself, the director field unwinds; however, due to the confined sample geometry the unwinding is not complete, and the residual twist of the director field persists even in the smectic phase. Another reason for the existence of the twist in the smectic A* phase is the increase in twist viscosity associated with the unwinding of the director field approaching the phase transition to the SmA* phase [19–21]. The continuous twist of the cholesteric phase, which is left-handed for both CN and CT, has the same sign as the residual twist of the director in the smectic phase. As a result the majority (80%) of the DHs examined are left-handed. The present authors believe that the opposite handedness of the remaining 20% of the DHs might have been the result of stresses in the layers caused by the aligning action of the hydrodynamic flow of the director during its unwinding during the phase transition.

The presence of hydrodynamic flows at the phase transition is clearly seen under the polarisation microscope. For strong planar azimuthal anchoring of the molecules at the substrates, the flows in the bulk of the sample can induce a twist (see the comment by Williams [4], p. 318) of handedness which depends on the direction of the flow; both chiralities are therefore feasible. Rheological properties of these materials have been studied experimentally by Asnacios *et al.* [17]. A continuous twist deformation is forbidden in the smectic phase; this residual twist of the director field relaxes through screw dislocations. It has already been mentioned that DHs are examples, but carrying a macroscopic Burgers vector.

A DH* is possibly a metastable distortion which relaxes the residual twist. The difficulty is that the helical axis of the cholesteric twist (which is perpendicular to the substrates above the transition) and the axis of a DH* (which appears parallel to the substrates below the transition) are mutually perpendicular. This fact suggests that the transformation of the cholesteric twist into a DH* is not direct but quite possibly goes through an orientational hydrodynamic instability. This instability appears in response to temperature quenching on cooling, if the time for which the sample is cooled below the transition temperature to the SmA* phase is less than the time needed for unwinding of the director twist existing above the transition. Such a situation is similar to that observed by Lonberg and Meyer [22] for lyotropic nematics twisted by an applied magnetic field which increases much more rapidly than the nematic director can twist. High viscosity associated with the twist deformation hinders the twisting of the director and as a result an instability takes place, yielding a periodic bend deformation for which the associated viscosity is much lower. In the case in which the director field unwinds on cooling when approaching the transition to the SmA* phase, the twist viscosity rapidly increases and the time needed to unwind the director becomes much larger than the time for the temperature to decrease below the SmA* transition [19–21]. It is therefore to be expected that the unwinding of the director is accompanied by an instability transforming twist to splay deformation, thus favouring the appearance of DH*s, for which splay is the main director deformation combined with the helicity of the structure.

3.2 Imperfections in double helical domains

Analysis of experimental observations of the DH textures shows that the double helical domains appear to be significantly distorted in both the SmA and SmA* phases. It is shown below that these distortions can be explained by a combination of at least three types of elementary imperfections. To introduce them it may help to briefly review the geometrical generation of an ideal DH.

If a straight line L is taken perpendicular to an axis Λ and gives it a helical motion along Λ with constant pitch p, the surface generated by L is a ruled helicoid, H. A family of equidistant layers parallel to H fills the space without multiple recovery in a region bounded by a circular cylinder, C (Figure 1), whose axis lies along Λ and whose diameter D_0 is related to the pitch p of H:

$$p = \pi D_0. \tag{2}$$

Outside C the focal surfaces of the parallel stacking intersect an infinite number of times (Figure 2(c)). It is seen that πD_0 is the perimeter of the circle contouring the cross-section of the cylinder C. When ruled on to a plane the surface of the cylinder C of height p transforms into a square with sides, $p = \pi D_0$. After ruling on to a plane the disclination lines within one period become straight lines parallel to one diagonal of the square. When the square is back folded into the cylinder, the projections of the disclinations on the plane containing the cylinder diameter will cross orthogonally, for an observer whose line of sight is orthogonal to Λ. This result is a particular case of the so-called Darboux's theorem, which concerns the geometrical nature of the focal surfaces of a family of parallel surfaces, and hence of a family of isometric layers. For example, the theorem states that the ellipse and the hyperbola of a FCD cross orthogonally along *any* line of sight, a result well known to Friedel *et al.* [6]. Therefore any violation of Equation (2) necessarily yields a violation of Darboux's theorem and indicates some deviation from isometry. Indeed, the cylinder surface becomes a rectangle with two different sides,

$p \neq \pi D_0$, and the disclinations become parallel to one of the rectangle diagonals when ruled on a plane. It is therefore clear that the projections of the disclination lines will cross at an angle other than 90° if $p \neq \pi D_0$.

Based on this generating procedure the following possible imperfections arise in the structure of the double helical domains.

3.2.1 *Asymmetric DH*

One of the imperfections most frequently observed in non-chiral SmA is illustrated in Figure 4, showing a DH in which one of the disclinations looks less wavy than the other, such that the two disclination lines have the same pitch but are a different distance from their common axis Λ. For this reason it is described as *asymmetric DH*. The analysis of the texture (Figure 4) suggests that this type of distortion corresponds to the case when the generatrix L of the central parent helicoid is tilted with respect to the axis Λ (see sketch in Figure 4). Smectic layers outside the DH are still perpendicular to the DH axis. It will be seen from Figure 4 that due to the tilt of the generatrix the number of the layers in two half-periods of the DH is different.

On the other hand a difference in the number of the layers in two half-periods can appear if an edge dislocation is superimposed on one of the two helical lines of the DH domain. Thus the generatrix L, tilted with respect to the rotation axis, can be a result of the

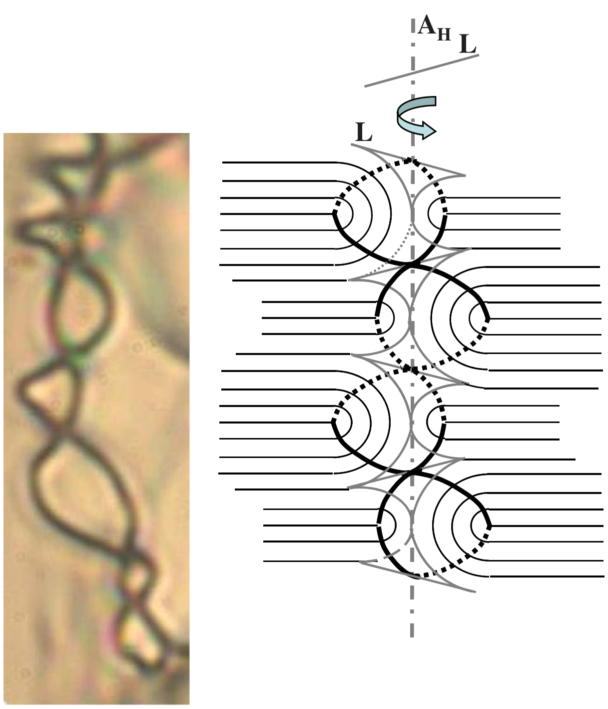

Figure 4. Asymmetric DH observed in 8CB, and a sketch of its structure.

interaction of an edge dislocation with the DH domain. This is represented in Figure 4, in which it is seen that the tilt of the parent layer generatrix is equivalent to the presence of an edge dislocation of Burgers vector b_e superimposed on one of the helices. In Figure 4, $b_e = \pm d_0$; the arbitrariness of the sign depending on which DH is taken as the ideal. Thus the number of smectic layers in the corresponding half-period of the DH is $n_2 = n_1 - b_e / d_0$, whereas in another half-period it is still n_1. The pitch of such an asymmetric DH is given by

$$p = (n_1 + n_2)d_0 = 2n_1 d_0 - b_e = p_0 - b_e, \qquad (3)$$

where p_0 is the pitch of some ideal parental DH. Taking into account the fact that a DH is equivalent to a screw dislocation, one can conclude that an edge dislocation superimposed on one of the two helical disclinations of the DH changes the Burgers vector of this screw dislocation by a value equal to b_e, depending on the sign in the following equation:

$$b = b_0 \pm b_e. \qquad (4)$$

It is seen in Figure 4 that $n_1 = 8$, $n_2 = 4$ and thus the pitch of the asymmetric DH is $|p| = 12d_0$. Difference in distances of the disclinations from their axes implies a deviation from Darboux's theorem, but in most cases this is not observed. Concerning the relation given in Equation (2), this cannot be checked directly due to ambiguity in determination of the diameter D_0 of the cylinder, which in this case is strongly distorted.

3.2.2 *Conical DH*

The next observation is that the pitch and width of the DH often vary along the DH axis. It is clear from Equation (2) that if, for example, p decreases along Λ, then D_0 should decrease proportionally, leading to a conical shape for the DH. Such a conical DH can be modelled as a sequence of coaxial cylinders of decreasing diameter. The links (thin lines in the insertion in Figure 5) between disclination segments (thick lines) belonging to the DH cylinders of different diameters are known as kinks [11]. Screw dislocations have to be attached to the kinks, as shown in the close-up in Figure 5. This type of DH distortion is observed in non-chiral smectics. The structure of the layers inside the conical DH can be represented as a system of equidistant layers parallel to a ruled helicoid generated by a generatrix L helically moving with the pitch varying along the axis Λ (see sketch in Figure 5). It is understood that the Darboux's theorem and Equation (2) are locally obeyed by this type of imperfection.

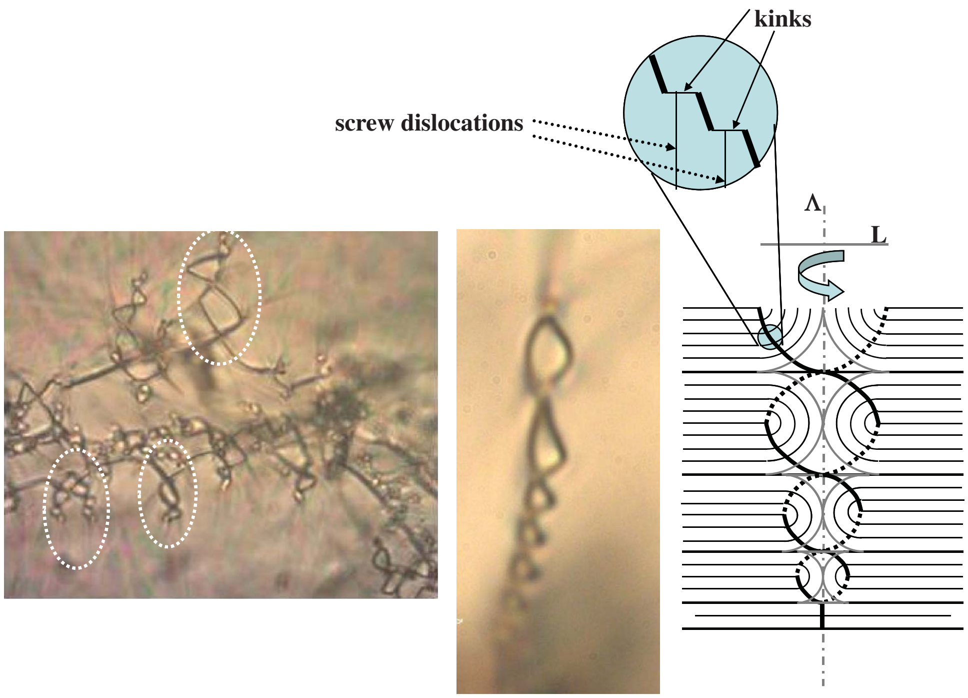

Figure 5. Conical distortion of a DH observed in the non-chiral SmA phase of 8CB and a sketch of the layers around the conical DH domain. Insertion shows the fine shape of the kinked disclination (thick segments), kinks (thin horizontal segments) and screw dislocations (dashed vertical lines) (colour version online).

*3.2.3 DH**

For double helical domains observed in chiral SmA*s we find that Darboux's theorem and the relationship between the pitch and diameter given by Equation (2) no longer hold. This is in strong contrast with the DHs existing in non-chiral smectics, in which both geometrical constraints are locally almost satisfied, even for imperfect DHs. This feature suggests that a DH* may exhibit specific imperfections different from those of a DH, as follows.

The layers inside the cylinder in an ideal DH are not twisted (a geometrical property of layers is to expel twist, because of the condition $\vec{n}.\overrightarrow{curl}\,\vec{n} = 0$), but clearly resemble a twisted geometry. A way of tracking the similarities and differences between the twist of a N* phase or a TGB phase and the ideal torsion of the layers inside the cylinder will be discussed in greater detail in a future paper, in which it will be shown that the ideal torsion of the layers results from the phase transformation of a N* fragment with the helical axis along the axis of the DH*, or a TGBA fragment with similar orientation. A certain number of screw dislocations can remain in the DH*s which form at the phase transition. These dislocations originate from the structural screw dislocations of the twist grain boundaries in the TGBA phase, or from a condensation of the twist of the N* phase. They connect two opposite kinks located on the two helical disclinations (Figure 6), and it is believed that bundles of such screw dislocations can be seen in Figure 7(a) and (b).

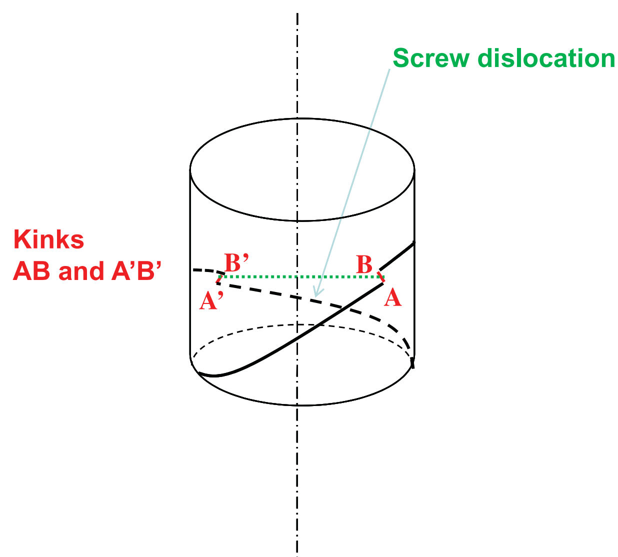

Figure 6. Schematic representation of the DH* cylinder with a screw dislocation attached to the disclinations at kinks AB, A′B′ (colour version online).

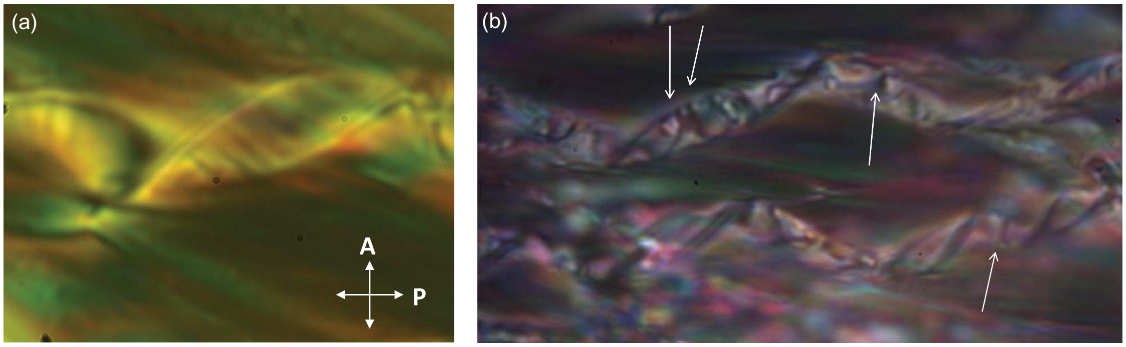

Figure 7. Bundles of screw dislocations (arrowed) connecting helical disclinations of the DH* in CN (a) and in CT (b).

3.3 Temperature behaviour of the DH domains

A further observation indicates that the pitch of the DH* is temperature dependent and decreases on cooling (Figure 8(a)). Figure 8 shows that on cooling, the DH* tends towards its ideal state; the Darboux rule becomes better obeyed and the pitch : diameter ratio tends towards π. The latter suggests that the temperature transformations of the DH*s on cooling consist in the elimination of screw dislocations from the DH* body. A segment connecting two disclinations seen in Figure 8 is a bundle of screw dislocations, which shorten on cooling such that at some moment the disclination lines cross and exchange by their segments, decreasing the pitch of the DH*, as shown in Figure 9. The reverse process in which pre-twisted nematic disclinations brought into contact cross, exchange by their segments and finally untwist has been experimentally documented by Ishikawa and Lavrentovich [23].

A decrease in the number of unit Burgers vector screw dislocations should lead to a weakening of the residual twist. If this is true, then the transformation of the pitch should not be reversible on further heating, which has indeed been observed (Figure 8). The irreversibility of the change in pitch on further heating indicates that the double helical disclinations are metastable distortions.

For non-chiral SmAs over a wide temperature range the DHs are only slightly sensitive to temperature but quickly disappear on heating towards the transition to the nematic phase. The observation reported by Williams [4] has been confirmed, that approaching the transition to the nematic state the DHs (quoting Williams) 'shorten in a non-continuous way by disappearance of the two segments forming half pitch each time' (Figure 10).

4. Conclusions

The present study has provided an empirical description of the characteristic features, as observed by light microscopy, of the double helical macroscopic defects (DHs and DH*s) present in SmA (made of achiral molecules) and SmA* (made of chiral molecules) phases. Both types can be related to an ideal domain model bordered by a set of two equal helical disclinations. But this ideal model is in reality never met with SmA and SmA* samples, although its physical existence is documented in the B7 phase of bent-core molecules, which is a lamellar phase of a completely different nature [5]. The ideal model of a DH or a DH* consists of a parent layer in the shape of a ruled helicoid (which can be geometrically generated by a straight line, described here as the generatrix, rotating helically about a fixed axis with constant pitch, p) on which are stacked parallel layers (isometry). This stacking stops short of a double helix with pitch, p, at a distance, $D_0/2$ of the axis ($p = \pi D_0$), and has to match with the layers outside the cylindrical domain just defined. The full geometry is equivalent to a screw dislocation of macroscopic Burgers vector $b = p$, as has been stressed by Williams [4]. This property is of course topological and is conserved when the DH or DH* is not ideal.

The discrepancies between DHs and DH*s and the ideal model are due to the interplay of the tendency towards isometry and the non-conservation of the layer curvatures at the border between the DH domain and the SmA matrix. This is complicated in the DH* case by the memory of the high temperature chiral phase (N* or TGBA) from which the DH*s are obtained when cooling to the SmA* phase.

It has been found that in the SmA* phase, the majority (approximately 80%) of the double helical disclinations have the same handedness as the residual twist remaining after the transition from the cholesteric phase; this is the same handedness as the molecular chirality. The double helical disclinations of the opposite chirality are most probably due to the twist induced by the hydrodynamic flows accompanying the unwinding of the director field during phase transition. The imperfections of the double helical domains can be described

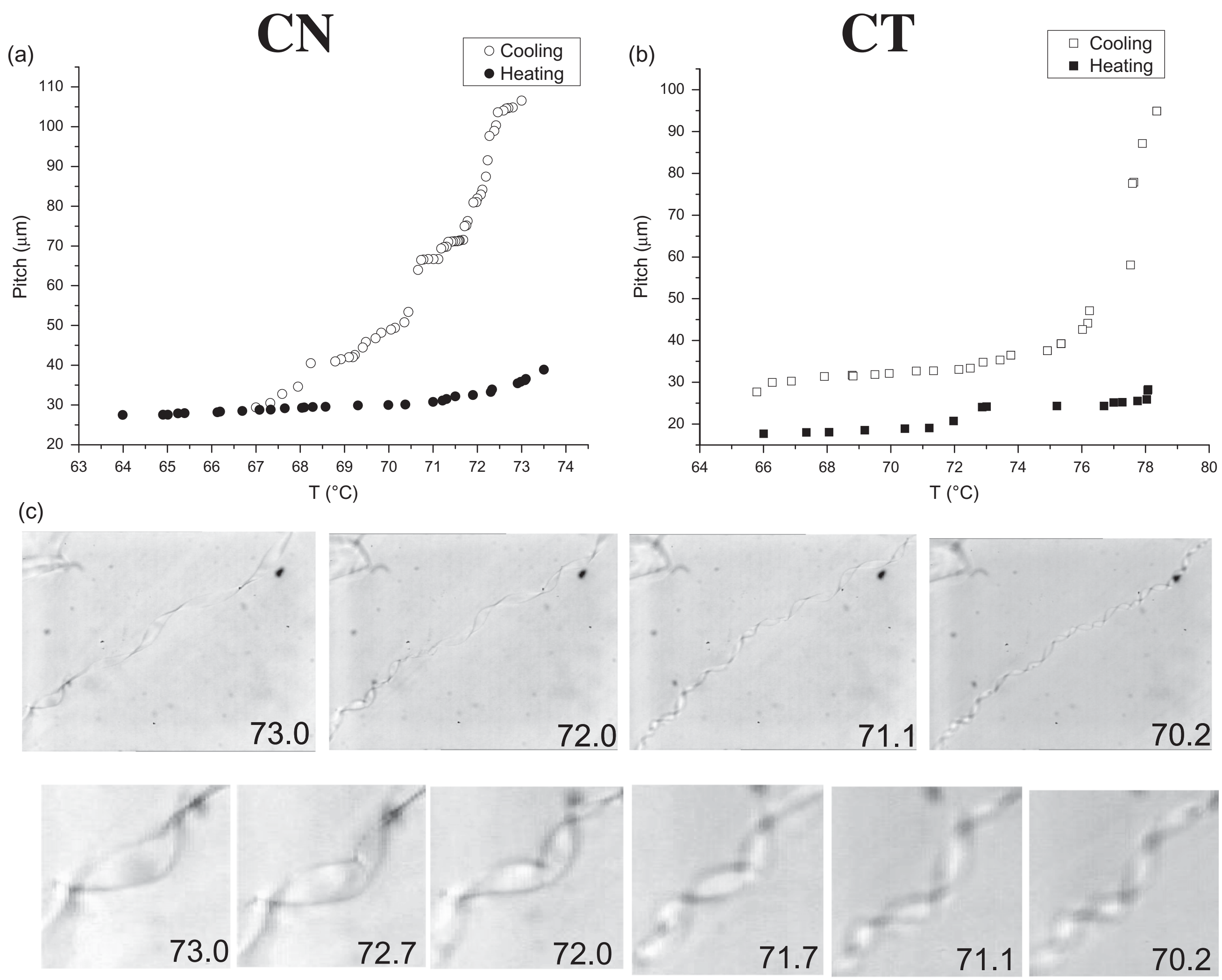

Figure 8. Temperature evolution of the pitch for CN (a) and (c) and CT (b). Numbers in right lower corner of the photographs in (c) from left to right display temperatures (°C) of the sample on cooling. The lower row of the photographs shows a magnification of the lower left corner of the corresponding photograph in the upper row.

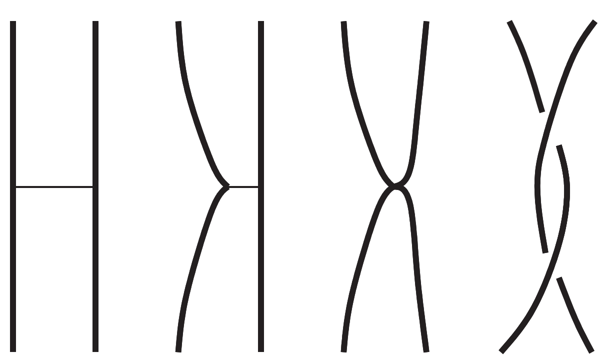

Figure 9. Crossing of disclinations leading to a decrease in the pitch.

as a combination of three elementary distortions of the central ruled helicoid, which can be classified in terms of imperfections in the rotation of the generatrix:

1. The generatrix is tilted with respect to the rotation axis. As a result the two helical lines have similar pitch but appear at a different distance from the rotation axis. This imperfection is frequently observed in SmAs.
2. The rotation rate of the generatrix is a function of its coordinate along the helical axis. The resulting DHs often have a conical shape. At such an imperfection the isometry is preserved, and the

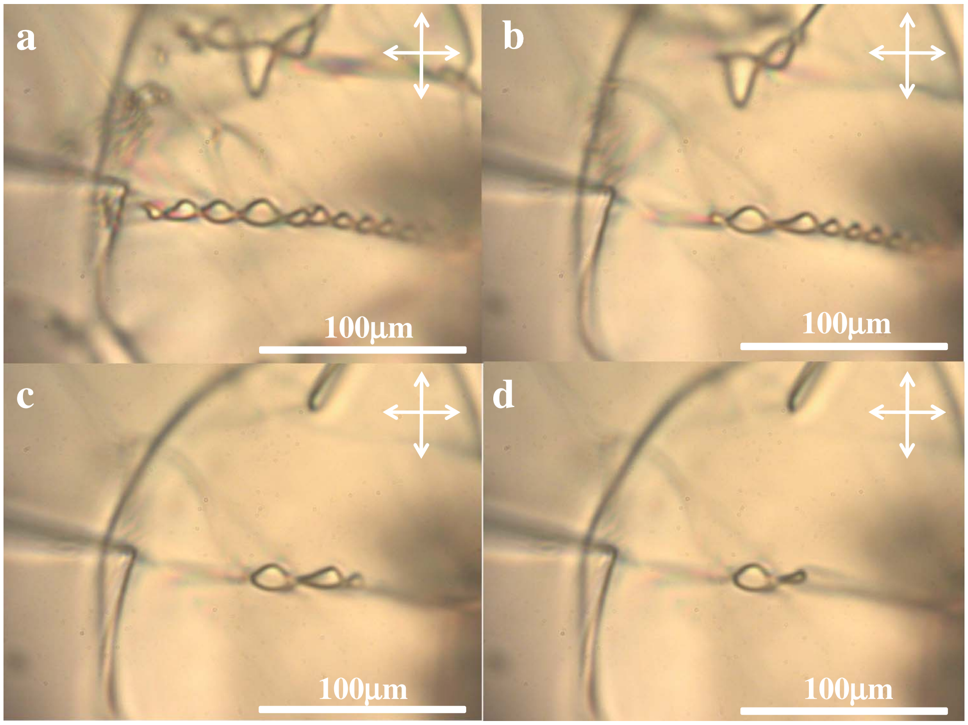

Figure 10. Temperature evolution of a DH in 8CB approaching the SmA nematic phase transition; thickness of the sample: 100 μm; the white arrows show the orientation of the crossed polarisers: (a) T = 33.00°C; (b) T = 33.05°C; (c) T= 33.28°C; (d) T = 33.3°C (colour version online). (Photographs: Vincent Andrieux.)

relationship $p/D = \pi$ holds. This type of imperfection is observed in SmAs.

3. Local breaking of the continuity in the helical rotation of the generatrix accompanied by screw dislocation segments orthogonal to the axis of the domain, crossing through it and connecting the helical disclinations. This yields an observable violation of the structural isometry. This type of imperfection is distinctly observed in SmA* but not in SmAs.

The pitch of the DH*s decreases on cooling. This decrease indicates that the Burger vector of the double helix decreases. On cooling, DH*s tend towards the ideal state, the Darboux rule is more closely obeyed, the pitch/diameter ratio tends towards π, and this transformation is irreversible on further heating. This suggests that the temperature transformations of DH*s on cooling consist in the elimination of screw dislocations from the DH* body.

A theoretical account of the question of helical defects in SmA and SmA* phases is in preparation.

Acknowledgements

The study was supported by the Ministry of Education and Science of Ukraine, as Project 0109U001062. YuN is grateful to the Université de Picardie Jules Verne for hospitality and financial support resulting from appointment as Invited Professor in 2007 and 2009. This is IPGP contribution # 2609 (MK).

Note

1. For example, the DH domains observed in the B7 phase [5] are ideal inside the cylinder (perfect isometry, the layers being parallel to the central ruled helicoid) but the outer part of the cylinder is distorted to the point at which it is in the isotropic liquid state.

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