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## Liquid Crystals

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### Quantum well/columnar discotic hybrids

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# Quantum well/columnar discotic hybrids

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Theory predicts that the deposition of a nanostructured dielectric film on the surface of a semiconductor quantum well will modulate its optical properties due to interactions between the quantum well exciton and the dielectric structure. We have chosen a columnar discotic triphenylene as dielectric medium since the columnar structure is able to provide spatial, and thus dielectric, modulations both on a 2 or a 0.4 nm scale, depending on the columnar orientation within the film. Film deposition on quantum well structures and model substrates by spincoating and via the vapour phase, in combination with annealing steps, gave rise to a rich spectrum of textural modifications and columnar orientations in the dielectric films. The investigations revealed significant modulations of the optical properties of the quantum well as a function of the textures and the columnar orientational order.

## 1. Introduction

Semiconductor quantum wells composed of nanoscaled layers display unique optical properties [1, 2]; because the confinement to thin layers gives rise to the formation of discrete sub-bands within the electronic band structure. Their location on the energy scale is controlled by the thickness of the layers in addition to the effects arising from the electronic properties of the specific materials from which the quantum well structures are prepared. One way to modify optical properties, such as the absorption and emission at constant chemical composition, consists in varying the thickness of the layers.

Another route consists in combining semiconductor quantum structures with photonic crystals characterized by a periodic modulation of the dielectric and optical properties [3, 4]. This combination gives rise to modulation of the Coulomb potential of charged particles in the quantum structure. Such combinations which are of interest, among others, for laser applications have already been considered experimentally [5–7]. This route was recently investigated theoretically in some detail by Eichmann *et al.* [8, 9], who considered a quantum well in close neighbourhood to dielectrically structured media such as photonic structures. The prediction was that the optical transition characteristics of the quantum well structure will be shifted by the presence of the dielectric structure. The sign of this shift is expected to depend on the relative magnitudes of the

dielectric constants of the quantum well material and the dielectric structure. The maximum of the excitation transition, as well as the band edge, are predicted to be shifted to larger values on the energy scale if the dielectric constant of the dielectric structure is smaller than that of the quantum well and *vice versa*.

Photonic crystals are structured on a scale amounting to several 100 nm [8, 9] which is larger by at least a factor of 10 than the spatial extension of the exciton within the quantum well. The Bohr radius of the Wannier-exciton is typically of the order of 10–20 nm in the quantum well structures considered in this paper [10]. It seemed of interest to us to study the effect of a dielectric medium on the optical properties of the quantum well for the case that the dielectric material is structured on a scale roughly one order of magnitude smaller than the extension of the exciton, i.e. of the order of 1–2 nm.

Such a structural scale is available, for instance, from columnar discotics [11–14], provided that the columns are oriented homeotropically with respect to the surface of the quantum well layers (figure 1). It is the intercolumnar distance which defines the scale of the spatial variations of the dielectric properties in this case. Yet the specific advantage of discotic liquid crystals in such applications is that a planar orientation of the same columnar system can be used to induce dielectric modulations on a scale which is smaller by another order of magnitude, i.e. 0.3–0.4 nm. Furthermore, one is able to induce in discotic systems textures which are homogeneous on a rather large scale on the surfaces. In order to control the optical properties of hybrids composed of quantum wells and columnar discotic

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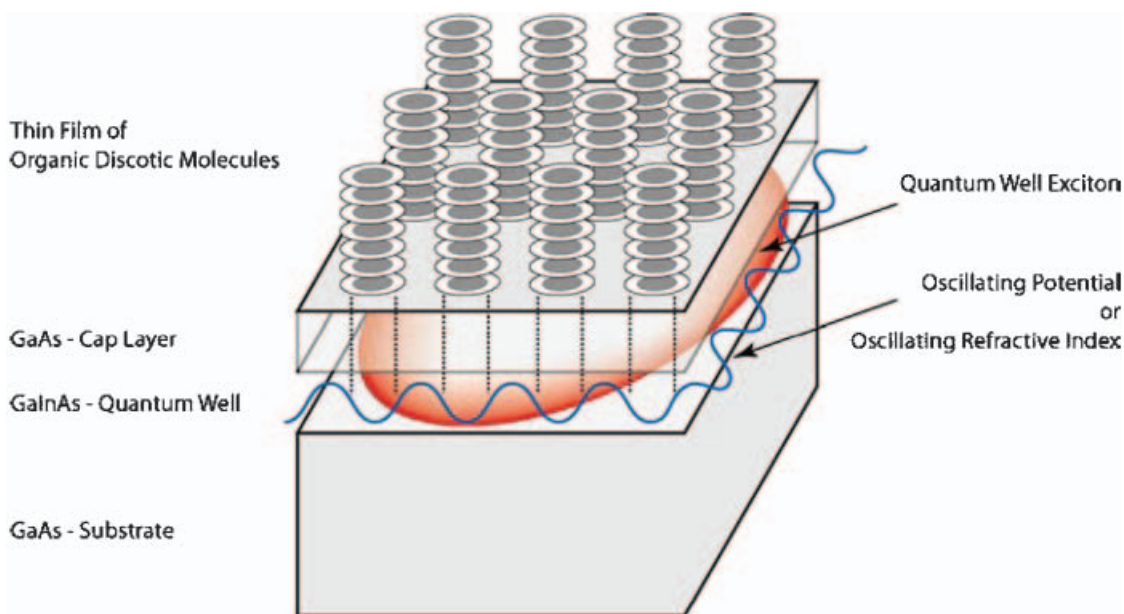


Figure 1. Arrangement of discotic columns on quantum well structures.

dielectric layers deposited on top of the quantum well structures, one must prepare thin discotic layers with well defined orientational states of the columns.

This paper is concerned with the structure displayed by such thin discotic layers composed of a triphenylene derivative deposited onto quantum well surfaces by vapour deposition (evaporation) as well as via spincoating from solutions. The paper furthermore reports on the structural modifications which set-in as the films are annealed at elevated temperatures, and it compares these findings with similar findings obtained on quartz and silicon surfaces as substrates. Finally the effect of the discotic dielectric film and its orientational state on the optical properties of the quantum well structures are discussed.

## 2. Experimental

Quantum films were prepared via a metal-organic vapour phase deposition of a 10 nm thick layer of (GaIn)As layer on top of a GaAs wafer. Within this layer 10% of the gallium atoms were replaced by indium. To isolate the quantum structure with respect to the electronic surface states, cap layers composed of GaAs having a thickness of 10 or 40 nm were evaporated on top of the layer structure. The band gaps of GaAs and of Ga<sub>0.9</sub>In<sub>0.1</sub>As amount to 1.55 and 1.44 eV, respectively and the Bohr radius characteristic of the quantum well structure amounts to 15 nm [10].

As columnar discotic material we selected the model compound 2-adamantanoyl-3,6,7,10,11-pentabutoxytriphenylene (AdaPBT, figure 2) which transforms on

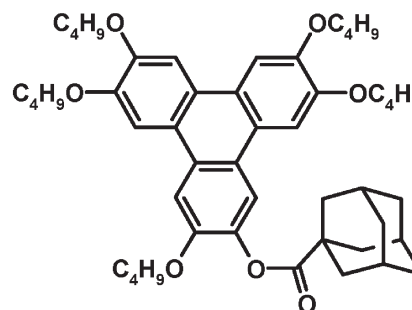


Figure 2. Chemical structure of the triphenylene compound AdaPBT.

cooling from the isotropic to the hexagonal ordered phase at 188°C, to a plastic columnar state at 168°C, and which finally is frozen into the corresponding glassy state at 85°C [15–17]. The hexagonal columnar phase is characterized by a three-dimensional registry of the centres of the disc-shaped molecules, as in a crystalline state. Yet the molecules are rotationally disordered with respect to the columnar axis. The X-ray diagram of the bulk material is characterized by sharp reflections at smaller scattering angles arising from the packing of the columns in space. The (110) is the most prominent reflection. A broad halo is observed at larger scattering angles superimposed by a set of reflections arising predominantly from the packing of the molecules along the column. A doublet is the most prominent feature in this scattering range (figure 3).

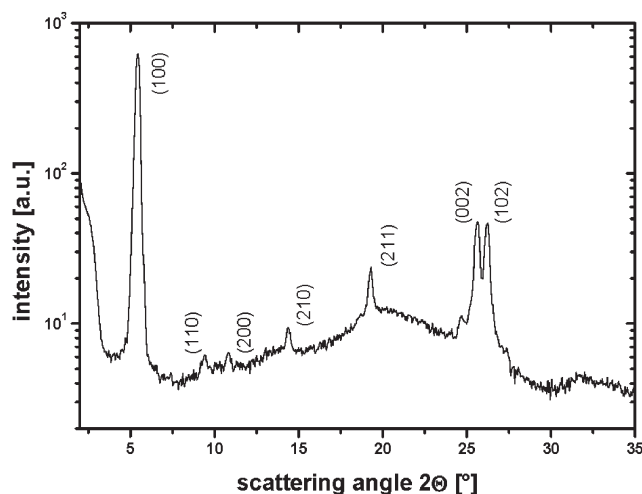


Figure 3. X-ray diagram obtained in the symmetric reflection mode characteristic of AdaPBT in its plastic columnar state.

The discotic films were deposited onto the GaAs quantum well structures as well as on model substrates, quartz and silicon. The substrates used in these studies were characterized by contact angle measurement on measuring system G 10 (Krüss). The measurements showed effective surface energies amounting to  $33 \text{ mN m}^{-1}$  for GaAs,  $39 \text{ mN m}^{-1}$  for the silicon substrate and  $43.5 \text{ mN m}^{-1}$  for the quartz substrate. The concept was that the structures formed in thin films on the substrates might depend on their surface free energy. To deposit thin discotic films onto the quantum well structures, as well as on model substrates, we used as one method spincoating starting from a solution with analytical grade chloroform as solvent. Typical film thicknesses that can be achieved in this way range from about 10 nm to several 100 nm. The films were all prepared from solutions with concentrations of 1 wt% using a spincoating apparatus (Convac) at a rotation speed of 2000 rpm for 60 s. The resulting films had thicknesses between 80 and 100 nm. The films were dried in a vacuum oven at  $70^\circ\text{C}$  for several hours to remove residues of the solvent. In a second approach we prepared discotic layers by vapour phase deposition using a high vacuum vapour deposition apparatus (Balzer PLS 500 E) at a source temperature close to the transition temperature into the isotropic phase ( $180^\circ\text{C}$ ) and at a pressure of  $10^{-6}$  mbar, with the substrate at room temperature ( $23^\circ\text{C}$ ). The film thickness achieved after one hour of deposition time was about 100 nm. A vibrating quartz set-up was used to control the layer thickness in this case.

The X-ray analysis was performed in the reflection mode using a Siemens D 5000 instrument with a copper target (wavelength  $1.542 \text{ \AA}$ ). Structural features were

further evaluated using optical microscopy (Axiotech and Labor Lux 12 Pol. S, both from Leitz). UV-vis and fluorescence spectroscopic studies were performed with an optical set-up built by ourselves.

### 3. Results and discussion

#### 3.1. Discotic films after deposition

Both spincoating and vapour phase deposition were controlled in such a way that thin films with a homogeneous thickness of close to 100 nm were deposited on the quantum well surface (GaAs) as well as on the model substrates quartz and silicon. The X-ray analysis obtained from the discotic films in the reflection mode displayed the prominent (100) reflection at smaller scattering angles but not the doublet at larger scattering angles. This observation strongly points to a planar orientation of the columns independent of the preparation method and independent of the substrate used. The films are transparent in the case of the quartz and the silica substrate but display a deep blue colour if located on the GaAs substrate. The blue colour is definitely not the result of an intrinsic absorption process characteristic of the discotic material but rather the consequence of thin layer interference effects. The UV-vis absorption spectrum displayed in figure 4 shows a broad apparent absorption peak located around 600 nm in addition to the absorption peaks between about 200 and 350 nm resulting from optical transitions in the triphenylene.

The columnar orientation resulting from spincoating and vapour phase evaporation onto substrates has been investigated in detail for pure (110) surfaces of gold as well as for the transparent electrode material ITO for a

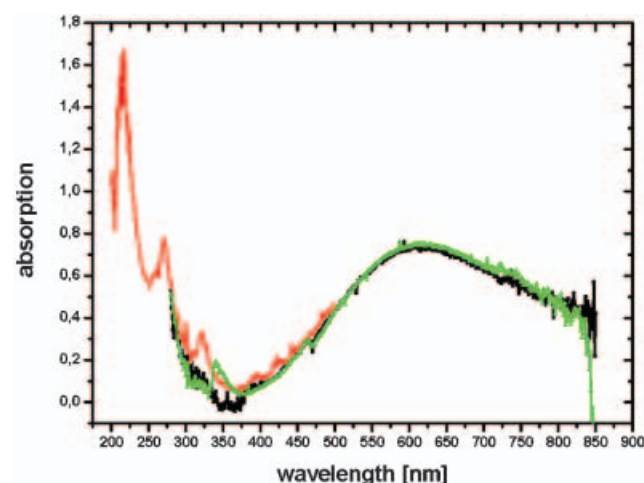


Figure 4. Superposition of different absorption spectra of the discotic AdaPBT film on a GaAs substrate taken in the reflection mode.

columnar discotic triphenylene [18]. The experimental finding was that the flat triphenylene molecules became arranged in a homeotropic fashion close to the substrate surface. This state of orientation changes with increasing distance, in the sense that the molecular axes are rotated by  $90^\circ$  yielding a planar arrangement of the columns. This happens in a transition layer amounting to about 10 nm, so that a planar columnar orientation prevails in films having a thickness of the order of 100 nm or more as considered in this contribution.

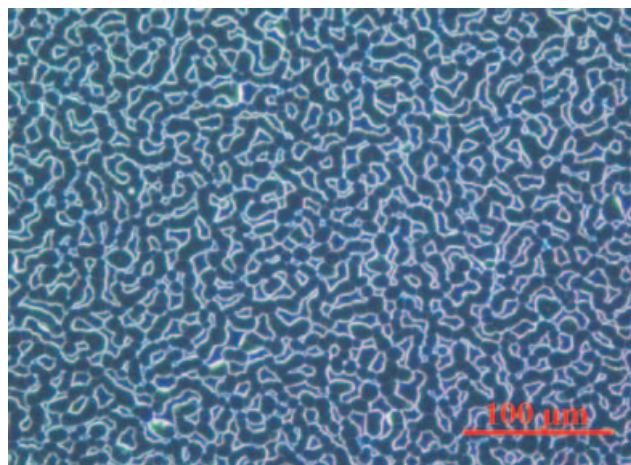
### 3.2. Annealing at elevated temperatures

Annealing at elevated temperatures is found to give rise to structural changes which are controlled predominantly by two different processes, namely dewetting and columnar reorientation. Dewetting is known to happen either via nucleation and growth or via a spinodal type of process [19–21]. Dewetting does in both cases result from statistical modulation of the thickness of the film. Which type of process, i.e. dewetting or reorientation, dominates and which type of texture results from these two processes depends significantly on the substrate on which the discotic layer is located. It is for this reason that we will discuss the results of annealing studies for the individual substrates.

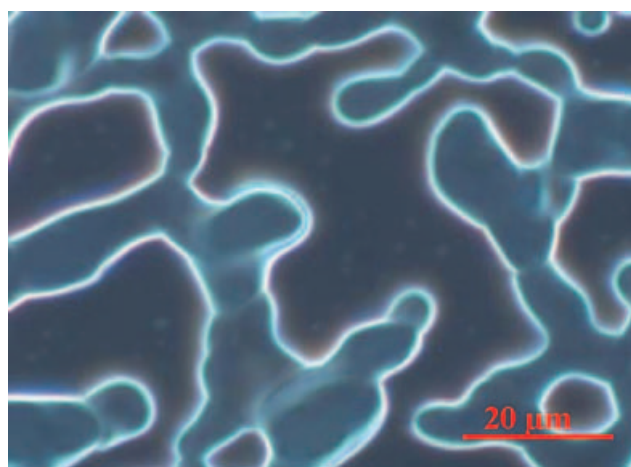
### 3.3. Model substrates quartz and silicon

Annealing at elevated temperatures in the neighbourhood of the transition into the isotropic phase leads to dewetting processes. An interesting observation is that the tendency towards dewetting is controlled to a certain extent both by the method used to deposit the discotic film and by the annealing conditions, i.e. whether annealing takes place in normal atmosphere or in vacuum. Vacuum and vapour phase deposition tend to increase the resistance towards dewetting, and one only observes the formation of cracks within the homogeneous films as nucleating sites for the dewetting process. Dewetting at later stages yields predominantly drop-like regions with irregular shapes (figure 5), resembling closely those observed for binary fluids after a spinodal decomposition followed by partial coarsening. It thus seems that dewetting happens predominantly via the spinodal process.

For an isotropic fluid state the expectation is that the final shape of the droplets after coarsening is circular, as controlled by surface free energy considerations. Yet discotic liquid crystals are structurally anisotropic, causing different planes within the columnar structure



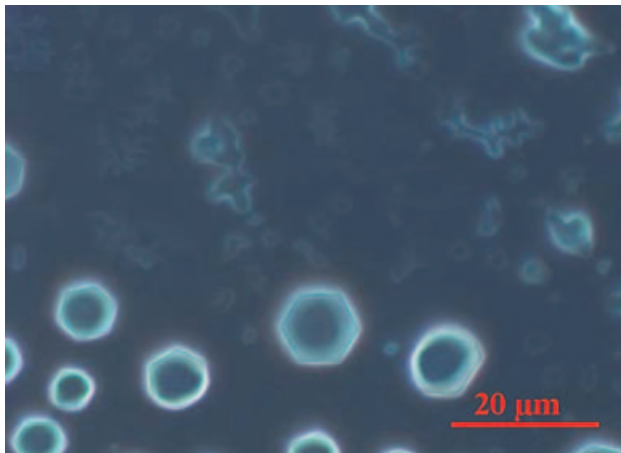
(a)



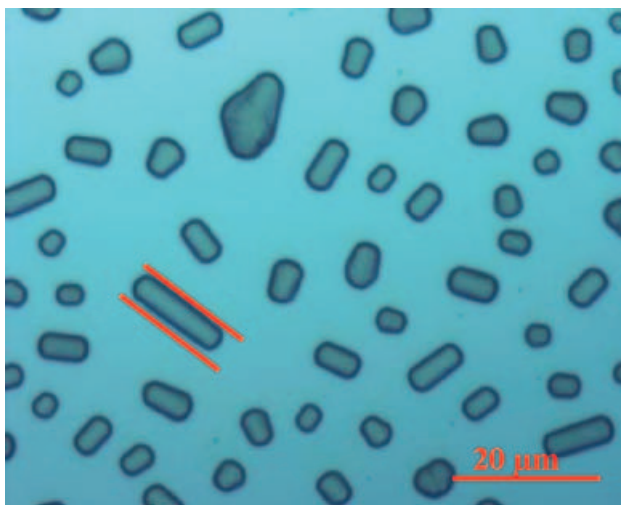
(b)

Figures 5. (a b) Dewetting structures: displaying regions with irregular shapes.

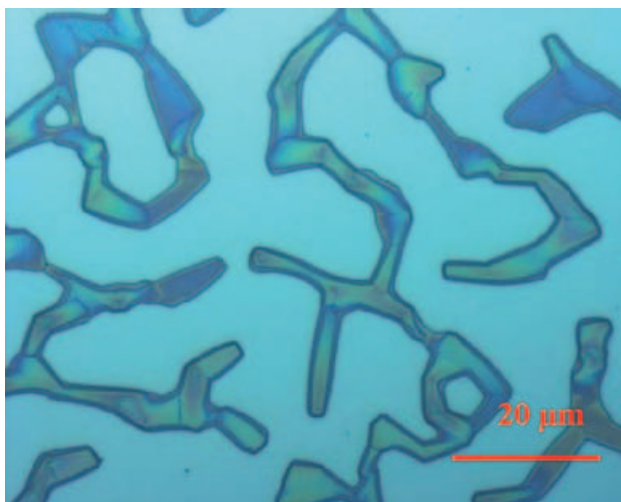
to possess different magnitudes of surface free energies. One consequence is that discotic droplets should have a tendency, even within the fluid-yet-liquid crystalline state, to display non-circular shapes; indeed, we observed the formation of hexagonal droplets due to dewetting, figure 6(a). Such hexagonal droplets have been discussed by us previously for various situations, and the finding was that their appearance is frequently connected with a reorientation of the columnar axis towards a homeotropic state [22–24]. The hexagonal shape thus reflects the two-dimensional hexagonal spatial structure characteristic of the columnar packing in a homeotropic arrangement. X-ray diagrams obtained on the annealed films are actually in agreement with this conclusion, they display the doublet reflection at larger scattering angles in addition to the (1 1 0) reflection.



(a)



(b)



(c)

Figures 6. Surface structures resulting from annealing (a) on silicon, (b, c) on passivated GaAs.

### 3.4. Passivated GaAs surfaces

GaAs surfaces were passivated with sulphur in order to reduce the disturbance of the optoelectronic properties of the quantum well structures by electronic surface state effects. Annealing of discotic films located on such surfaces also gave rise to the onset of dewetting processes, yielding droplet-like regions with irregular shapes as discussed above. Yet, rather than showing hexagonal droplets as a final state, cylindrical droplets with the caps being structured in a hexagonal shape were observed, figure 6(b). We have to conclude that these types of dewetting structure reflect spatial structures characteristic of a planar arrangement of the columns. Dewetting is clearly not correlated with columnar reorientations in this case, and the X-ray results are in agreement with this conclusion. The X-ray scattering diagrams display the (1 1 0) reflection but no doublet at larger scattering angles.

### 3.5. GaAs substrates

Annealing of the homogeneous dark blue discotic film located on the GaAs surface gave rise to two different types of textural and structural change depending on the annealing conditions. Annealing well within the temperature range of the transition into the isotropic phase resulted in peculiar dendritic textures to be discussed below in more detail.

Annealing close to but outside the transition range, i.e. in a range where the discotic structure is still stable, gave rise to dewetting yielding irregularly shaped droplet domains similar to the cases discussed above for the model substrates. This type of textural change will be discussed briefly at first. It appears that two types of such droplet structure occur simultaneously: one in which the droplets are formed by a discotic film located on the substrate, and one in which this structure is formed by voids occurring within the otherwise homogeneous discotic film. This is not surprising since one would expect such inverse structures based on the cocontinuity of spinodal structures. In any case these dewetting processes do not cause a reorientation of the columns from planar to homeotropic orientation, as evident from the X-ray diagrams.

This is in contrast to the case of annealing well within the transition range. The X-ray diagram clearly shows that the majority of columns reorient in this case and assume a homeotropic orientation. The intensity of the otherwise very prominent (100) reflection is strongly reduced, whereas the wide angle reflection is not only detected but has a significantly larger scattering intensity than the (100) reflection (figure 7). An interesting feature is that the wide angle reflection is

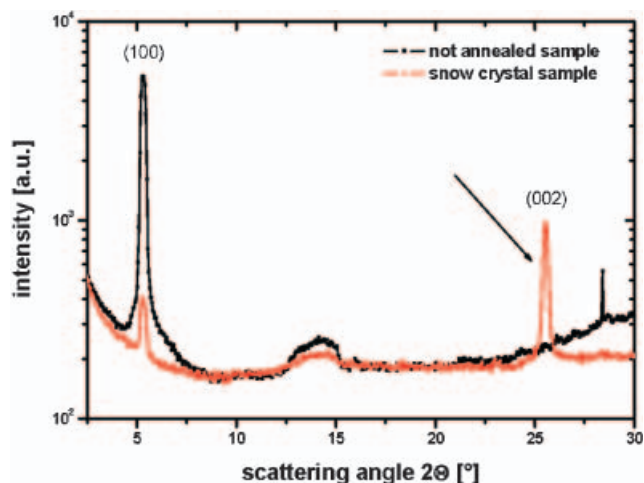
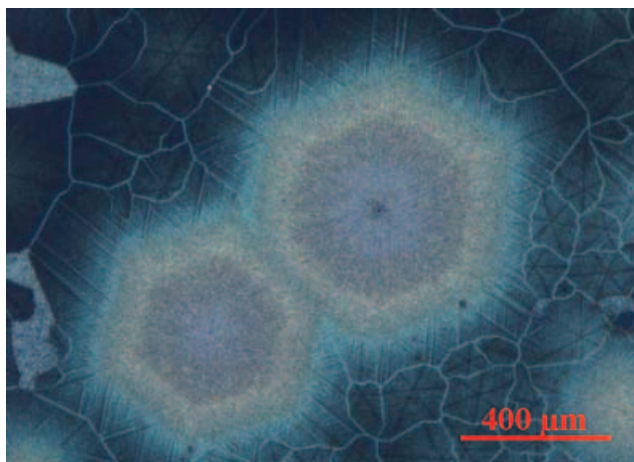


Figure 7. X-ray diagram obtained for an annealed discotic film on GaAs.

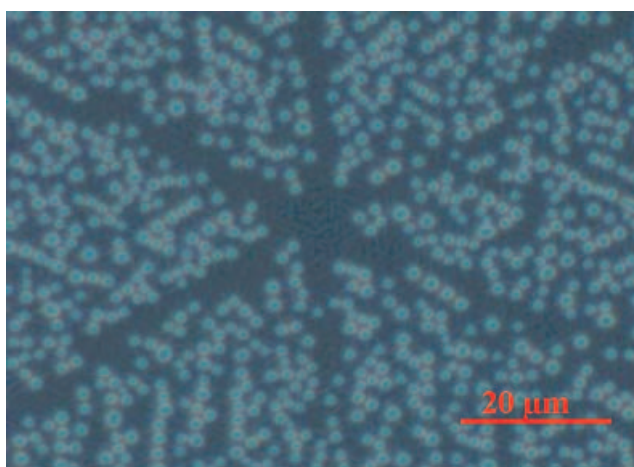
no longer a doublet but rather a single peak, indicating that the hexagonal columnar phase rather than the plastic columnar phase has been frozen in.

The textural features observed for films annealed in this way are rather complicated and characterized by various length scales. One observation is the formation of domains with a length scale of up to the mm-range with narrow bright domain walls, figure 8 (a). Within the domains very bright hexagonal regions with dimensions in the several  $100\mu\text{m}$  scale form, the brightness apparently resulting from scattering phenomena. An enlargement of such structures shows an intrinsic hexagonal symmetry with well recognizable symmetry axes pointing towards the edges of the hexagons, figure 8 (b). The elements which form such structures are hexagonal regions with a size of the order of  $1\mu\text{m}$ , with a dendritic type of arrangement following the hexagonal symmetry [21].

More detailed information is available from AFM studies (figure 9). These reveal that the hexagonal regions are in fact voids reaching down to the substrate surface. Yet these studies also reveal that the voids within the surrounding discotic film are connected by valley-type structures, also following the overall hexagonal symmetry. These structures are not homogeneously distributed within the discotic films but modifications are observed which may be earlier and later stages of the texture formation process. A possibly earlier stage is one in which only valley-type structures have been formed within the otherwise homogeneous film, and a later stage is one in which the voids have grown, losing their hexagonal shape and assuming an irregular drop-like shape as observed in dewetting, figure 9 (c).



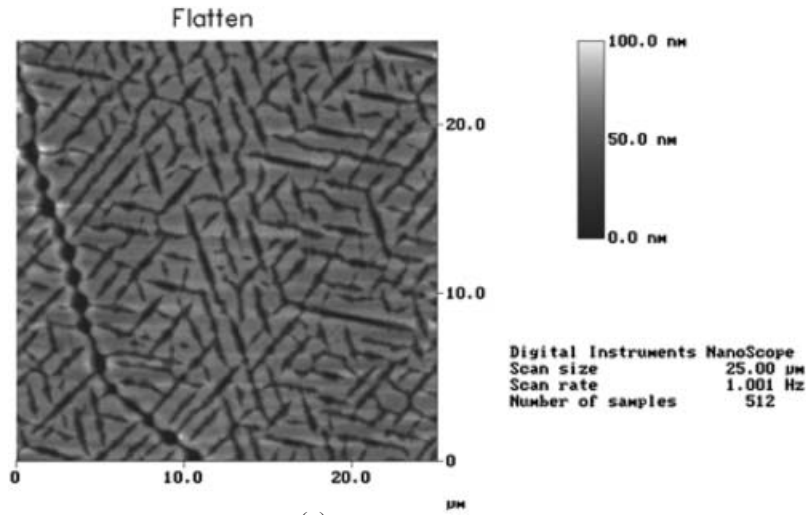
(a)



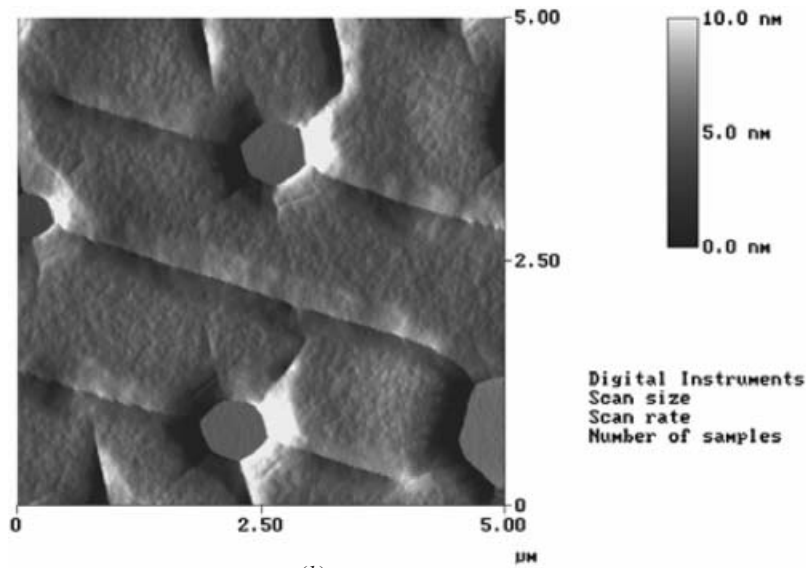
(b)

Figures 8. (a, b) Textures formed by annealing a discotic layer well within the melting range on GaAs on different spatial scales.

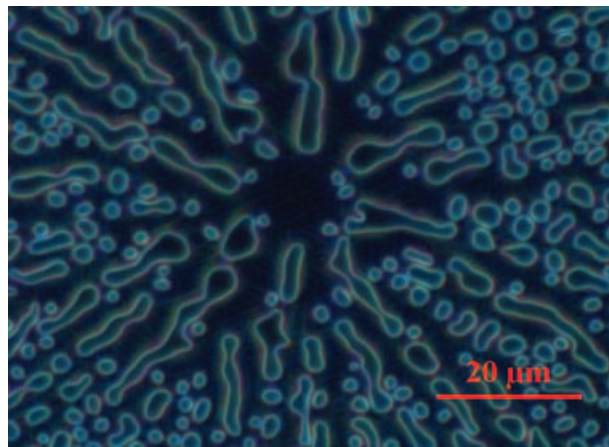
At present we do not have a convincing model which could explain the complex textural features observed experimentally. In any case the hexagonal symmetry, which is displayed by the texture on all spatial scales, indicates that annealing at temperatures within the transition range to the isotropic phase apparently gives rise, in a first step, to a discotic film in which the columns have become homeotropically aligned. Yet this film undergoes structural changes as the temperature is decreased. Domain walls are formed probably due to mechanical stresses. Furthermore valley structures as well as hexagonal holes are formed which can grow and change their shape indicating the presence of transport processes. Our tentative interpretation is that layers with the homeotropically aligned columns start to grow in thickness, starting at particular nucleation points and extending along directions defined by the hexagonal



(a)



(b)



(c)

Figures 9. (a, b) AFM results, (c) optical results on textural features of discotic layers on GaAs.



symmetry of the packing of the columns. The transport of columnar material to the areas growing in thickness gives rise to a depletion of material in neighbouring regions. This, in turn, gives rise to the formation of the valley structure and finally of the hexagonal holes. The regular arrangement of hexagonal holes and valley structures is again controlled by the original hexagonal columnar lattice structure.

### 3.6. Optical properties of the surface-modified quantum well structures

To evaluate the effect of the deposition of the dielectric discotic films on the optical properties of the quantum well structures, we investigated the fluorescence spectra. The emission of the quantum well without the dielectric layer is shown in figure 10, both for the cases of a 10 nm and a 40 nm thick cap layer. Each emission spectrum is characterized by a broad peak, with a maximum at  $1.406 \pm 0.001$  and  $1.409 \pm 0.001$  eV, respectively, and a width of 8 and 7 meV, respectively. The emission spectra are to a first approximation symmetric.

The deposition of the dielectric layers on the quantum well structures gives rise to several types of fluorescence modification. The peak maximum is shifted, the width increases and the emission peak shows in certain cases a broad shoulder at the low energy side. This is obvious from figure 11, displaying the spectra for quantum well structures with various types of dielectrically structured discotic films.

The as-deposited films, characterized by a planar orientation of the columns, give rise to a shift of about 4 meV of the peak maximum to higher energies. This is well outside the range of accuracy amounting to about 1 meV. The peak intensity is strongly reduced whereas

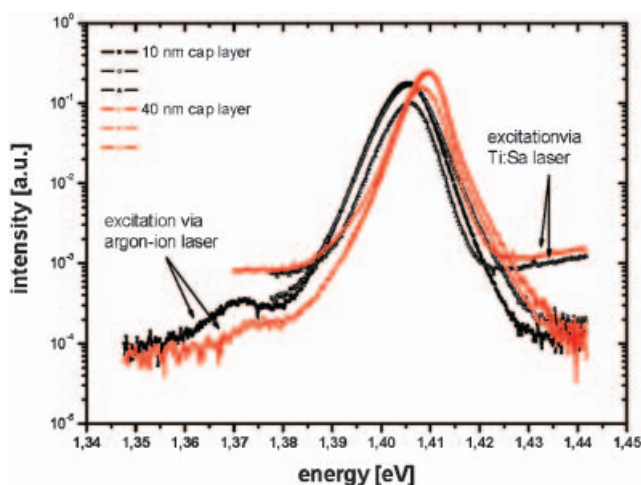


Figure 10. Fluorescence spectra of the quantum well structures without dielectric layers.

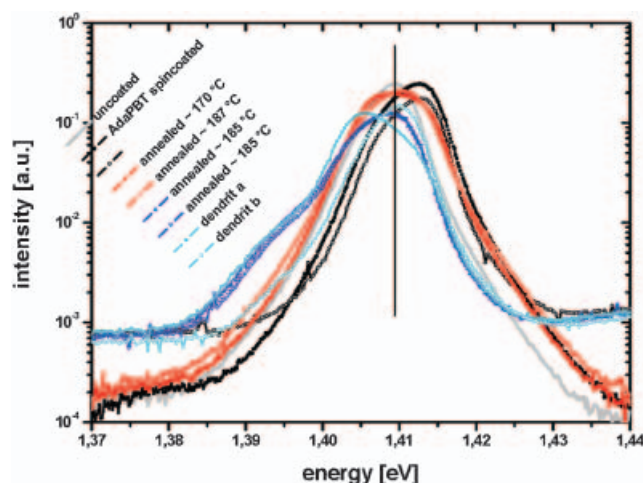


Figure 11. Fluorescence spectra of quantum well structures with various types of discotic films.

the peak width is increased in such a way that the total emission intensity stays about constant. This indicates that the deposition of the dielectric films and the modifications of the electronic properties induced in this way do not give rise to novel routes of energy relaxation or of energy dissipation. A partial reorientation of the columns, as induced by annealing, gives rise to a shift of the peak maximum to lower energies relative to the uncoated quantum films by about 3 meV and, again, to a reduction of the peak intensity and an increase of the peak width. Dielectric films displaying a partial reorientation of the columns furthermore give rise to a low energy shoulder on the emission peak. Similar modifications are introduced by the dielectric layer for quantum wells with passivated surfaces. The shifts of the emission maxima are somewhat larger than in the case of the unpassivated quantum wells, amounting to up to 6 meV.

These results show that a dielectric film deposited onto a quantum well structure causes a modulation of the optical properties of the quantum well even for the case that the structural modulations within the dielectric film take place on a structural scale, which is one or two orders of magnitude smaller than the extension of the excitons in the quantum well. At present we are unable to offer interpretations for all the features described above. Yet it seems that basically the theory put forward by Eichmann [8, 9] can account at a qualitative level for the modification of the emission properties. The basic concept is that the optical properties of the quantum well structures are modulated due to a dielectric coupling of the quantum well and the dielectric layer. The exciton defines a polarization which induces a mirror polarization within the dielectric layer. The mirror polarization, in turn, modulates the

interaction between the electron and the hole and, thus, the opto-electronic properties of the exciton and the corresponding emission properties.

Furthermore it seems that the dielectric situation is such that a planar orientation of the columns gives rise to an effective dielectric constant within the dielectric film which is smaller than that of the quantum well, and a homeotropic orientation gives rise to a dielectric constant which is larger than that of the quantum well. This would account for the different signs of the shift of the peak maximum relative to the pure quantum well structures. A further tentative interpretation is that the shoulder observed for the partially reoriented films is the result of inhomogeneous orientational states within the films. It is known from studies on columnar discotics in confinement that transition layers occur between different states of orientation displaying lower degrees of order [25, 26]. More detailed experiments and theoretical work will be needed to interpret the optical properties properly.

### Acknowledgement

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