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Influence of Surface Relief on Orientation of Nematic Liquid Crystals: Polyimide Doped with WS_2 Nanotubes

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Abstract: Among the different methods for orienting liquid crystal (LC) molecules, adding nanoparticles into the matrix of the substrate material towards modifying its surface, is actively pursued. In this context, the influence of the nanoparticle content on the texture of the surface of polymer film used as the substrate for the LC orientation is of particular interest. Thus, in the current paper, WS_2 nanotubes were used to dope the polyimide (PI) substrate-film in order to modify and control its surface morphology/roughness and properties. The modified organic surface structure is applied in order to achieve a new means for controlling the orientation of the LC molecules. This tool adds to the classical methods for controlling the orientation of the LC molecules, such as the display technique.

Keywords: nematic liquid crystals; orientation relief; sensitization; contact angle; novel way for liquid crystal display technology

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1. Introduction

It is well-known that the doping of organic material by nanoparticles (NPs), i.e., sensitization, can dramatically influence its fundamental properties [1–10], including the perspective structures based on nematic liquid crystals (NLC), used generally in display technology, optical limiting schemes, solar energy harvesting devices, biomedicine, etc. [11–15]. In particular, it is important to understand the effect induced by the NPs on the NLC properties and on the structure of their interfaces.

Many types of NPs were considered for this objective. Fullerenes, carbon nanotubes, quantum dots, shungites, reduced graphene oxides, lanthanides NPs, Janus NPs, WS_2, SiO_2, TiO_2 NPs, etc., have been used as dopants in NLC matrices. Introducing nanoparticles into the LC mesophases were shown to stimulate supramolecular organization and photoinduced electron transfer in the LC media [16]. It also led to the modification of the refractive parameters of the liquid crystal cells used for holographic recording [17–21], prompting the stability of the liquid crystal phases [22] and increasing the polarizability and conductivity of the LC structures [23–25], etc. Moreover, the relief at the interface between the LC mesophase and the solid substrate can have a dramatic effect on the basic features of the LC structure, especially its orientation [26–30]. This relief can be induced, for example, by a photo alignment process, which leads to the formation of some polymer networks or by changing the content of the nanoparticles in the organic substrate film. It should be noted that different authors reported that the relief structure can affect the orientation of the LC molecules at the interface from planar to tilted and to homeotropic ones.

In the current paper, we continue the study of the substrate relief structure in order to find new ways to orient the LC structures. As an indicator for the change in the orientation of the LC molecules induced by the NPs, the variation of the contact angle is considered for the system consisting of polyimide (PI) materials doped with WS_2 nanotubes (WS_2 NTs). Previously WS_2 NTs with the different content were used to dope the LC mixture in order to increase their polarizability and refractive index via the mechanism proposed in [23]. Moreover, by adding the nanotubes, the switching time (switch-on and switch-off parameters) was improved, thus, the speed of the LC cells with the dopant based on WS_2 NTs was increased. Therefore, the correlation between the spectral, structural and switching characteristics of the LC cells doped with WS_2 NTs were established. These data were firstly published in the papers [31,32].

2. Materials and Methods

Photosensitive layers of polyimides with the previously studied chemical formula [33,34] were used. This layer was sensitized with WS_2 nanotubes (NTs), which were chosen for this purpose due to their high aspect ratio, mechanical strength and semiconductive nature. Such sensitization provided significant influence on the contact angle. The synthesis and properties of WS_2 nanotubes have been scrupulously studied in the past [35–37].

Figure 1 presents a schematic rendering of the process for the step-by-step preparation of the polymer films and control of the LC molecule orientation by the relief structure. Here, 1.5% polyimide solution in tetrachloroetane was prepared, to which 0,1 wt.% WS_2 NTs were added. The WS_2 NTs-doped polyimide film was deposited on a glass substrate made of K8 crown material and dried for 12 h at room temperature. A schematic illustration of the orientation of the LC molecules is shown as the green oblate-shaped fragments overlaid on the atomic force microscopy (AFM) image of the relief (right-down). A scanning electron microscope (SEM) image of WS_2 nanotube powder is shown in the top right of this figure.

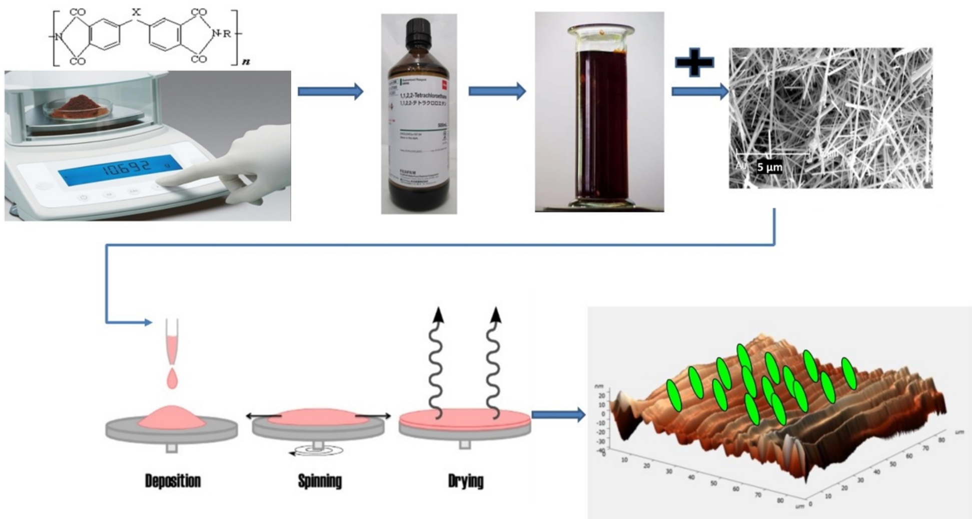

Figure 1. Schematic illustration of the process for preparing the relief structure of the PI film surface with WS_2 nanotubes as dopants, and the subsequent orientation of the LC molecules (green oblate features overlaid on the surface relief on the right-hand side).

The OCA 15EC set-up purchased from LabTech Co. (St. Petersburg-Moscow, Russia) was used to measure the wetting angle (contact angle) at the doped organic film surface. Additionally, the modified surface was analyzed using a Solver Next (AFM) atomic force microscope (purchased from NT MDT Co., Zelenograd, Moscow Region, Russian Federation). The AFM instrument was operated in semi-contact mode in air-atmosphere.

In order to check the orientation of the LC molecules on the proposed relief, an LC mixture of 4-pentyl-4-biphenylcarbonitrile, 98% (Aldrich Co., Karlsuhe, Germany), was used. Additionally, in analogy with the investigation of WS_2 nanotubes, doping of the polyimide substrate was done with single wall carbon nanotubes (SWCNTs), type #704121 (Aldrich Co.). Their effect on the orientation of the LC molecules was studied as well.

3. Results and Discussion

Table 1 summarizes the results of the wetting angle measurements for the current samples and for the benefit of comparison with films studied before.

Table 1. Variation of the wetting angle α with the nanoparticles added for the currently studied surfaces in comparison with different organic thin films used previously for this aim.

Material Drop	Organic Film Material	Sensitizer Type	Sensitizer Content %	Film Thickness (μm)	α before Sensitization, °	α after Sensitization, °	Ref.
Water	PI	C_{70}	0.5	3	72	89	[38]
Water	PI	C_{70}	1.0	3	72–73	103	[39]
Water	PI	WS_2	0.1	3	94	104	current
LC *	PI	WS_2	0.1	3	16	29	current
Water	PI	CNTs	0.1	~3–4	75–79	101	current
LC	PI	CNTs	0.1	~3–4	30	34	current
Water	PMPS **	C_{60}	0.83	4	75	81	[40]
Water	PVA	C_{60}	0.1	50	40	83	[40]
Water	PVA	CNTs	1	50	39–40	82	[40]
Water	NPP	C_{60}	1	3	97	102	[40]
Water	PNP	C_{70}	1	3	90–91	94	[40]
Water	PBMA ***	C_{60}	0.34	2.5	54	61	[40]

* 4′-Pentyl-4-biphenyl-carbonitrile (Sigma-Aldrich, Karlsuhe, Germany). ** PMPS—poly(methyl phenyl silane). *** PBMA—poly(butyl methacrylate).

Analyzing the data shown in Table 1, one can testify that the wetting angle is increased significantly after the sensitization of the polymer film with the WS_2 NTs. The same tendency can be established for the CNTs used for the polyimide sensitization. As shown in Table 1, this effect is not limited to the nanotubes and occurs also for other nanoparticles incorporated into the polyimide matrix. Let us briefly discuss, for example, the fullerene C_{70}, which influences the relief. The fullerene C_{70} has the form of a rugby ball structure and it forms a fairly uniform distribution of molecules in the organic matrices. In addition, it should be taken into account that C_{70} has, basically, greater electron affinity energy than an *intra*molecular acceptor fragment. This allows it to form *inter*molecular complex with charge transfer to the polymer in the matrix quite efficiently. The remnants of C_{70} molecules can reside on the polymer surface and manifest their ridge on the surface.

Thus, concerning the surface modified with the WS_2 NTs, this modification can be reasoned by the fact that part of the WS_2 nanotubes serve as intermolecular acceptors, interacting with the donor moiety of the conjugated organic materials based on polyimide, poly(methyl phenyl silane), poly(butyl methacrylate) groups. This donor–acceptor interaction provokes a novel reorganization of the polymer lattice and formation of extra free volume in the nanocomposites. Furthermore, a fraction of the NPs, reside close enough to the polymer surface and can affect surface relief, which is manifested through the variation of the contact angle of the liquid droplets. The contact angle with neat and doped polyimide surface relief is shown in Figure 2. Here, Figure 2a,c show the contact angle for a neat polyimide surface, while Figure 2b,d show the contact angle in the case of the polyimide surface doped with the WS_2 nanotubes. The variation in the contact angle due to the doping was estimated using water drops (Figure 2a,b) and LC drops (Figure 2c,d).

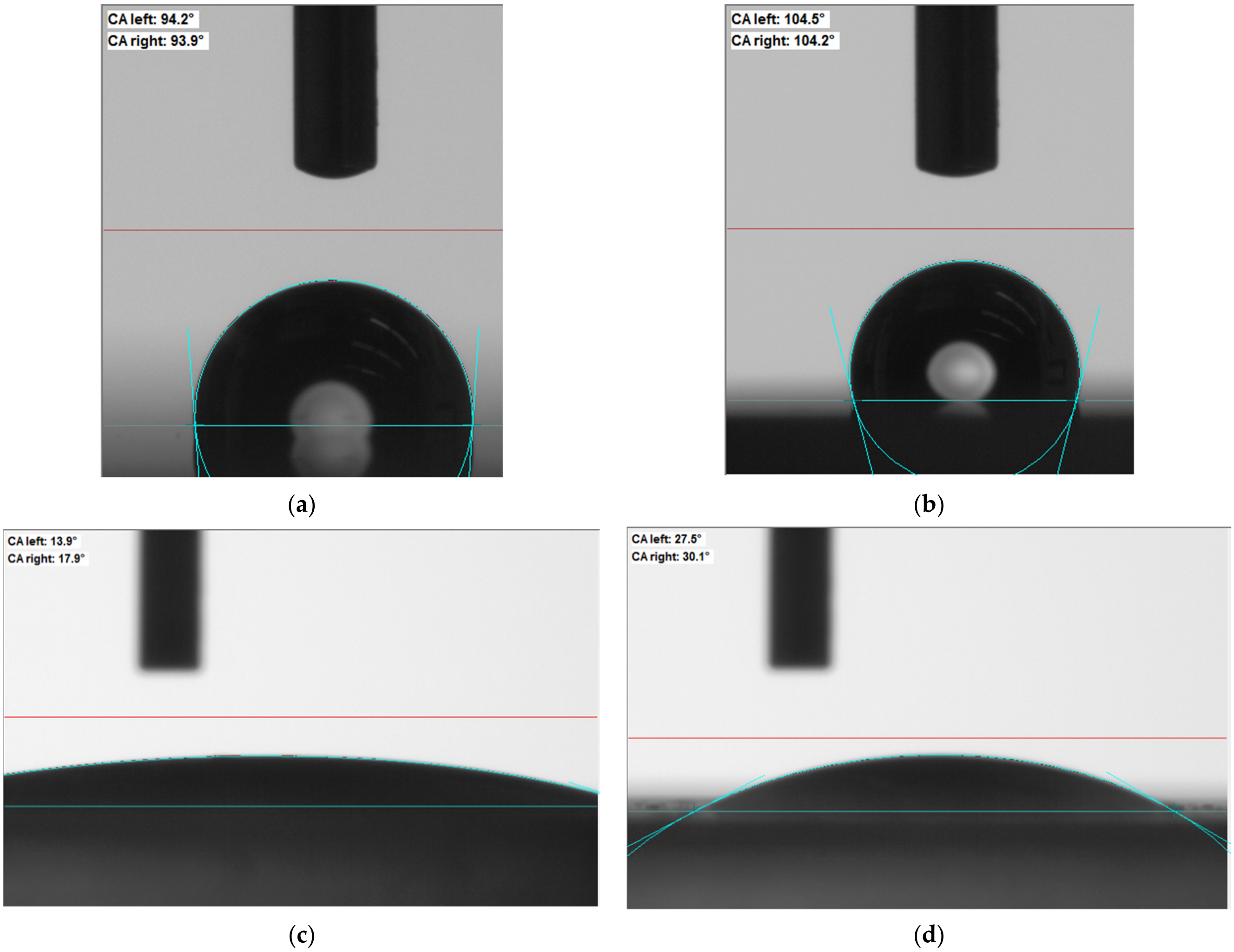

Figure 2. Wetting angle at the surfaces of: (**a**) pure PI (water drop); (**b**) PI with WS_2 NTs (water drop); (**c**) pure PI (LC drop); (**d**) PI with WS_2 nanotubes (LC drop).

One can see that after polyimide sensitization by the WS_2 nanotubes, when water drops were used, the contact angle increased from ~94 degrees up to ~104 degrees, and from ~16 degrees up to ~29 degrees, when LC drops were applied on the PI surface. It can be anticipated, therefore, that the orientation of the LC molecules at the polyimide surface can be modulated by varying the content of the NPs, and WS_2 NTs in particular, in the organic matrix film. Thus, different orientations of the LC molecules can be potentially obtained, namely, from the planar position, to the tilted and homeotropic ones. It should be noted here, that this presented method to orient the LC molecules allows modulation of the LC molecule orientation in addition to the classical methods of aligning the LC molecules at the interface in order to create the LC cell in the *S*-, *B*- or *T*-mode. Figure 3 illustrates the use of the classical approach to modulate the orientation angle of the LC molecules, which can be effectively supported by the contact angle measurements.

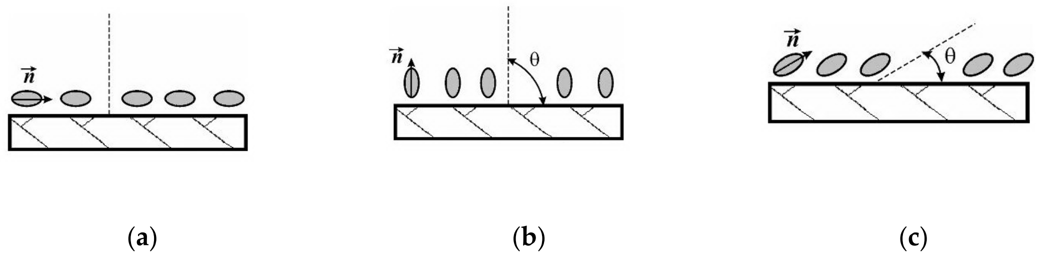

Figure 3. Illustration of the modulated orientation of the LC molecules in the planar (**a**), homeotropic (**b**) and tilted (**c**) position.

To support the idea, that the contact angle as well as the orientation of the LC molecules can be modified by doping of the polyimide, AFM analysis of the pure polyimide surface, and one, which was doped with the WS_2 NTs, has been made. The data are presented in Figure 4. One can verify that the period of the observed grating Λ^{-1} at the surface is two times larger for the PI structured with WS_2 NTs compared to the neat PI surface.

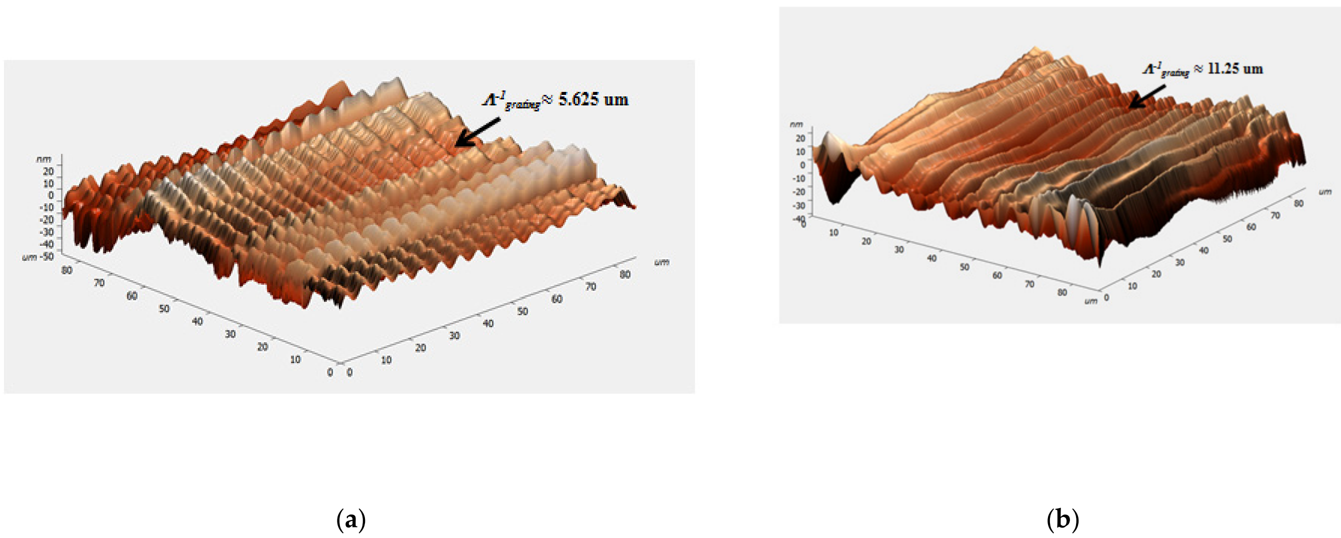

Figure 4. The relief obtained on the pure polyimide surfaces (**a**); the relief obtained at the WS_2 NPs-doped polyimide surface (**b**). The dimensions are equal for both images.

The relief parameters of pure PI and PI doped with WS_2 nanotubes ($90 \times 90\ \mu m^2$) is shown in Table 2. It should be emphasized, that different methods to test the material surface roughness can show somewhat different results, but the relative change due to the WS_2 NTs doping of the polyimide surface is likely to be of similar magnitude. It is interesting to observe that the WS_2 NTs can increase the surface grating pitch (please see data from Table 2). This effect may be attributed to the fact that the WS_2 nanotubes are stretched along the polymer lamellas, changing their intermolecular spacing, increasing thereby the pitch.

Table 2. Summary of the relief data for pure and WS_2 NTs-doped PI matrix.

Surface Type	Root-Mean Square Roughness (S_q), nm	Average Roughness (S_a), nm	Maximum Area Peak Height, nm (S_p)	Maximum Area VALLEY Depth, nm (S_v)
Pure PI	11.284	8.796	32.149	47.889
PI + WS_2	8.480	6.648	24.880	41.674

One could notice from Figure 4, that the average periodicity of the pure polyimide surfaces (Figure 3a) and the relief obtained at the WS_2 NTs-doped polyimide surface

(Figure 3b) are different. While the pitch distance is larger, the relief structure itself is finer, i.e., the roughness and the area of the pitch are smaller for the WS_2 NTs film. This effect can be attributed to the fact that the WS_2 nanotubes can be placed in the film, not only in same direction of the polyimide lamellas, but also in an orthogonal direction refining thereby the relief structure. Possible consideration of this phenomenon is illustrated in Figure 5. The WS_2 NTs are placed inside the polyimide matrix in different directions. Indeed, the effect of the WS_2 NTs arrangement in the organic film, as well as their concentration, should be studied in greater detail in connection with the relief structure of the PI film in the future. Other novel 3D local volume features can be possibly established on the polymer film via the doping process, which will be studied by SEM analysis, as well as a host of other techniques in future studies.

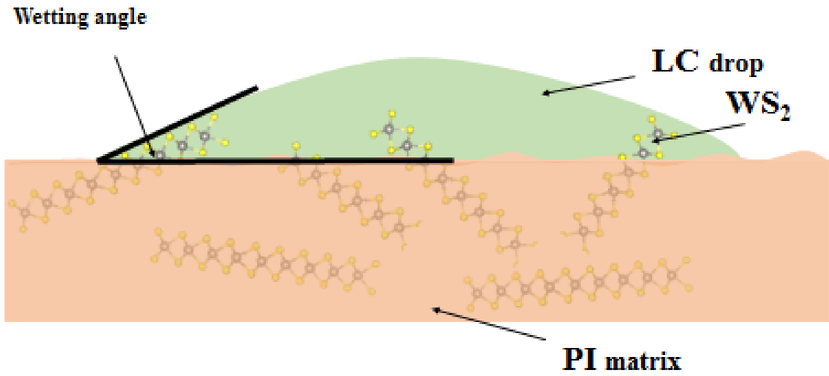

Figure 5. Schematic illustration of the WS_2 NTs protruding from the PI matrix and their influence on the surface texture and the LC drop orientation.

Thus, it can be noticed that the doping of the polymer with nanotubes modifies the structure of the organic films. Therefore, the sensitization process of the polyimide film can be used to control the orientation process of the LC molecules. It can extend the range of methods to align the LC structures, e.g., for optoelectronic device applications. Indeed, varying the content of the nanoparticles in the organic matrix of the planar, homeotropic or tilted orientation of the LC molecules can be obtained and modulated according to the NPs content, which suggests a novel orientation method for display technology. It should be also mentioned that this process can be used for optical limiting. In this kind of application, the reflection and the diffraction effects should be considered as additional mechanisms to attenuate the irradiated light.

4. Conclusions

In summary, organic polyimide films doped with WS_2 nanotubes were proposed in order to align the LC molecules in different directions.

AFM images and contact angle analysis were carried out to visualize the effect of the WS_2 NTs on the structure of the surface relief. Comparison with other polymer matrix materials doped with fullerenes C_{60}, C_{70} and CNTs, is presented. The collected data extended the range of applications of the doped polyimide systems. Technologies such as display devices, optical limiting and biomedicine can potentially benefit from this approach in addition to the classically used TN (twist nematic), IPS (in plane switching) and MWVA (multi walls vertical alignment) technologies used in optoelectronics and medical technologies.

It is possible, though too early to clearly state, that the method of varying the angle of inclination (orientation) of LC molecules by varying the concentration of the nano-objects inside the orienting matrix base, accomplished here via doping of polyimide films with tungsten disulfide nanotubes, will simplify all currently available methods of orienting LC molecules for display and modulator technology. This approach is expected to reduce the number of technological operations required for the fabrication of LC-based optoelectronic devices since the photosensitive matrix base will be able to act not only as a recording layer in the modulation device but also as an orienting electro-optical layer for reading the information.

For future studies, the nature of the substrate should be taken into account as well. In the current experiments, neutral glass substrates have been used in order to eliminate the effect of the symmetry of the materials. In practice, however, crystalline materials such as KBr, ZnSe, Si, Ge, etc., are used as substrates in the electrically- and optically-addressed liquid crystal spatial light modulator area, for example.

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Data Availability Statement: The data presented in this study are available in [29,38].

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