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Investigation on the Vertical Alignment Transition by UV-Irradiation after Slit-Coating of UV-Curable Mesogen-Added Liquid Crystals

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SUMMARY The slit coater method is an excellent liquid crystal (LC) alignment control technique that can order the LC alignment even on plastic substrates without pre-forming optional LC alignment films. However, controlling an arbitrary pretilt angle is still one of the issues. To elucidate the essence of the mechanism of the alignment transition from the planer to vertical alignment by UV polymerization, an in-liquid atomic force microscope was introduced. As a result, it was deduced that the LC alignment transition is induced by the realignment of mesogenic groups rather than surface topological change.

key words: *UV-curable mesogen, slit coater, surface orientation, surface energy, atomic force microscopy*

1. Introduction

The technical demand for flexible electronic display devices is not only durability against physical shock. For example, as displays are increasing in size, the emergence of novel wall-mounted displays that are foldable and packaged in advance and then unfolded after the product delivery is strongly expected [1], [2]. To realize foldable displays, replacing glass substrates with plastic substrates is essential. The reason why the foldable displays have not been launched in earnest until today is because the development of the plastic substrates is still in progress. Focusing specifically on liquid crystal displays (LCDs), maintaining the thickness and molecular alignment of the liquid crystal (LC) is a major issue when folding the LCDs [2], [3]. LC molecular alignment films to maintain molecular alignment on the substrate surface also have problems. Polyimide (PI) is the most common alignment film for LCDs, however, PI requires a baking process at high temperatures after coating on the substrate, and this thermal process may wrinkle the plastic substrate. Therefore, an LC alignment control technique that does not require an alignment film preparation has been desired. Previously, a novel liquid crystal alignment control method that does not require an alignment film named the 'slit coater method' has been demonstrated [4]–[9]. Several interesting physical properties have been discovered through a series of studies regarding the slit coater method whereas no manufacturing equipment in practical use utilizes this technology at present. One of the shortcomings is that the pretilt angle cannot be controlled well [7]. The original slit coater method was characterized by forming a planar alignment layer on the substrate surface by applying UV-curable mesogen-doped nematic liquid crystal (NLC) using a slit coater and simultaneously irradiating UV light. However, it has been confirmed that when UV is irradiated after liquid crystal coating is finished, LC molecules in the bulk realign vertically [4]. There is no doubt that the reaction of UV-curable mesogen [10] plays an important role while the cause of this phenomenon remains completely unknown. It is expected that a technique for controlling the pretilt angle will be devised if the essence of the mechanism of this alignment transition is elucidated.

In this study, we investigated the cause of the transition from planar to vertical alignment depending on the timing of UV irradiation. First, reproducing the orientation transition phenomenon using common NLC is confirmed. Second, we hypothesized and verified the hypothesis that the alignment transition was caused by a decrease in surface energy. Third, we used atomic force microscopy (AFM) to verify whether the polymerized UV-curable mesogens form a mesoscopic-level topological surface structure. These indirect pieces of experimental evidence will help us understand the essence of the orientation transition phenomenon. Although supplemental PI alignment film is not required, it is also not possible to control a pretilt angle.

2. Alignment Transition by Post-Coating UV Irradiation

Figure 1 illustrates the schematic diagram of the slit coater system including the slit nozzle and UV-light source [4]–[8], [11]. UV-curable mesogens mixture used is composed of acrylate derivatives doped with photo initiator (UCL-011-K1, DIC), which was added 1.0 *wt*% into the host NLC. The mixture as a coating solution is transfused into the slit nozzle by a syringe pump. The NLC mixture discharged from the slit nozzle is coated on the substrate with a uniform thickness of approximately 1~2 micrometers. Since LC molecules are planarly aligned by shear flow force, it is possible to instantly

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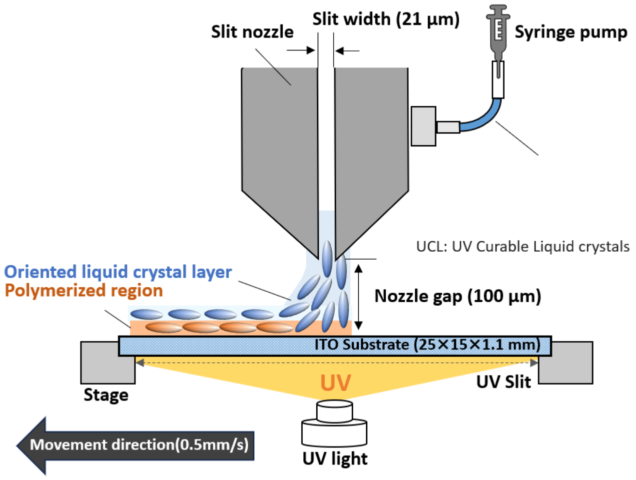
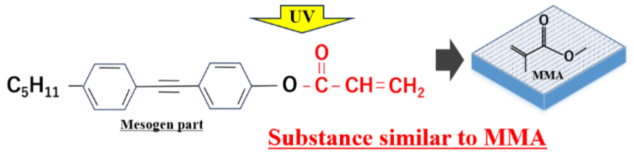
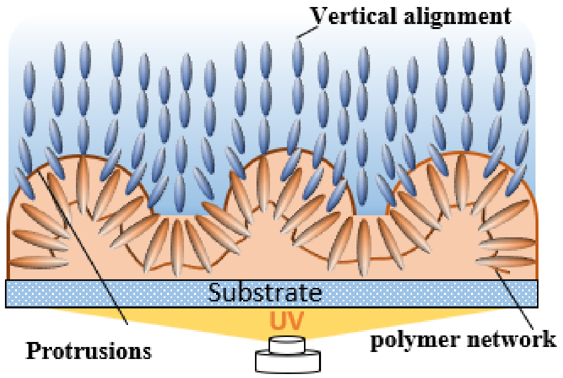


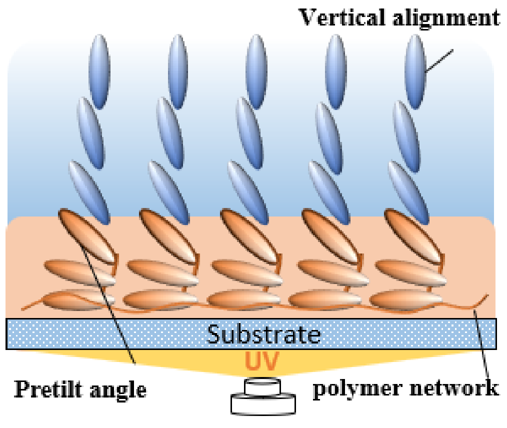
Fig. 1 Slit coater system and its schematic diagram.



(a) Surface energy decrease model



(b) Mesoscopic model



(c) Microscopic model

Fig. 2 Models of possible three factors of vertical alignment.

anchor the alignment planarly by irradiating them with UV light, as detailed in the previous paper. Aiming to further simplify the LC coating system, we attempted a process revision in which UV was irradiated after finishing the LC coating, and it was confirmed that the planar liquid crystal alignment was realigned to vertical LC alignment [4], [6], [7].

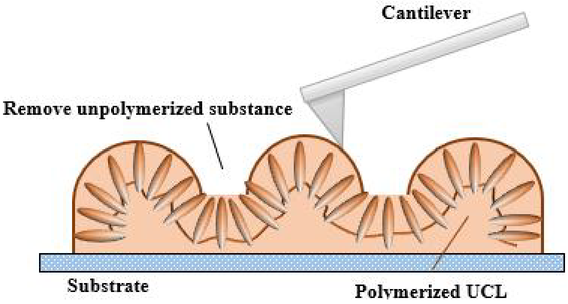
It is known that several factors cause liquid crystals to exhibit vertical alignment. Creagh and Kmetz described that substrates with surface energy lower than the liquid crystal surface tension cause vertical alignment [12]. Furthermore, it has been known for a long time that vertical alignment can be achieved by forming a topological shape on the substrate surface, such as in an obliquely evaporated silicon monoxide (SiO) film [13], [14]. Zhang *et al.* demonstrated a large pretilt LC alignment reaching 40° by lithographically fabricated topological structure [15]. Maeda and Hiroshima demonstrated that nano-sized porous structure provides vertical LC alignment [16]. It may also be effective to coat a thin film with functional groups that align LC molecules vertically. Recently, supramolecular comblike polyimides with mesogenic side chains for stable vertical alignment of LCs have been prepared [17]. It has also been reported that UV-curable mesogens segregated on the substrate by UV irradiation contribute to vertical alignment [18]. The possible cause of such alignment transition by post-coating UV irradiation seems to be any of the above three factors. Models of the above three factors are shown in Fig. 2. It is assumed that UCL-011-K1 (hereafter UCL) is a mixture of acrylate derivatives as shown in Fig. 2 (a) whereas the components are not disclosed [10]. The first hypothesis is that UV irradiation causes the methyl methacrylate (MMA) unit to separate and then polymerize to form a thin film, whose low surface energy causes the LC molecules to align vertically. The second hypothesis is that UV irradiation causes mesoscopic-sized protrusion, as shown in Fig. 2 (b). The third hypothesis is that the splay alignment occurs inside the UV polymerized layer, as is generally believed (Fig. 2 (c)) [19]. To elucidate the leading cause, in this study, we estimated the surface energy of the slit-coated layer surface and observed the surface topology by AFM before and after UV irradiation.

3. Experimental Methodology

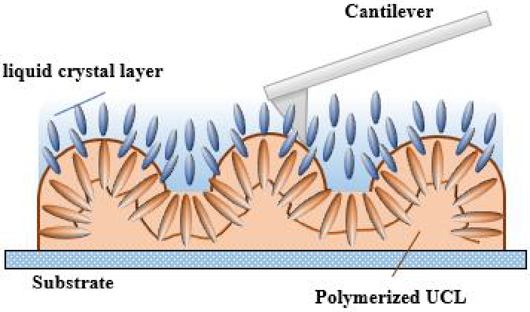
NLC mixtures as a host LC whose base component is fluorinated molecules (NTN-02, DIC) were used to observe the transition from planar to vertical alignment. The following two types of UCL mixed NLCs were coated on an Indium Tin Oxide (ITO) electrode on the soda lime glass: one sample was UV irradiated at the same time as the LC mixture coating (referred to as simultaneous UV irradiation), as shown in Fig. 1. Another sample was UV irradiated after finishing the LC mixture coating with a slit coater (referred to as post-coating UV irradiation). The glass substrate whose dimensions were 15×25 mm^2 was cleaned by the vacuum plasma asher (CUTE-MP/TD, Femto Science, Korea) before the LC coating process. The UV light source was SP-9 (Ushio Inc.), whose center wavelength was 365 nm and UV exposure intensity was approximately 10 mW/cm^2. The moving speed of the sample stage in Fig. 1 was 0.5 mm/s.

Surface energy was measured using the common contact angle method [11], [20]. To determine the surface energy, contact angle measurements on the surface were made by the sessile drop method with a goniometer DM-100 (Kyowa Kaimenkagaku Co., Ltd.). The test liquids used were ethylene glycol, glycerol, and pure water. For comparison, a sample in which an MMA (Wako, Co.) doped with 9 *wt*% of photo-initiator DMPAP (2,2-dimethoxy-2-phenylaceto- phenone, Aldrich) was spin-coated on glass instead of the UCL mixed liquid crystal was prepared.

The AFM instrument used was SPM-8100FM (Shimadzu Co.), and the cantilever was OMCL-AC160TS (OLYMPUS, Japan) whose specific values in air are as follows; the resonance frequency = 300 kHz, the force constant = 26 N/m, the length = 160 μm, width = 40 μm, and the thickness = 3.7 μm, respectively. A piezo scanner with a large range (X, Y: 30 μm, Z: 5 μm) was selected so that the mesoscopic scale topological structure could be observed. We observed the surface topology of the outermost surface of the UV polymerized UCL layer using the following two methods: the first is a conventional dynamic mode in an atmospheric environment. For this measurement, excess liquid crystal substance was removed after completing the UV polymerization. Although it is highly reliable as an AFM method, there is a concern that the outermost surface may be deformed when removing the liquid crystal. The second method is widely called in-liquid AFM [21]–[23]. In this method, the cantilever tip is immersed into the LC layer for observation without removing the LC layer above the UV polymerized surface. It seems that the latter measurement method can provide a real outermost surface of the UV polymerized UCL.

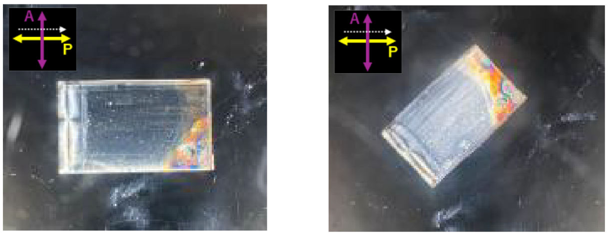


(a) conventional dynamic mode AFM

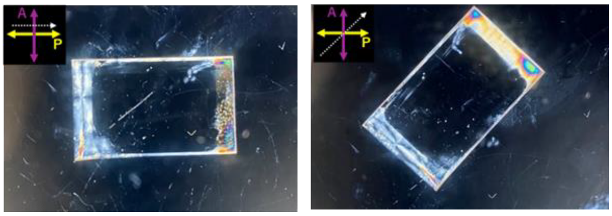


(b) dynamic mode, in-liquid AFM

Fig. 3 Models around the cantilever tip in two AFM measurement methods



(a) LC coating without UV irradiation



(b) post-coating UV irradiation

Fig. 4 Photographs of the alignment texture of slit-coated LCs depending on the timing of UV irradiation.

4. Results and Discussion

First, to reconfirm the planar alignment fabricated by the original slit coater method and the alignment transition from the planar alignment to vertical alignment caused by the post-coating UV irradiation [4], the LC alignment textures are represented in Fig. 4. All photographs were taken under crossed Nicols, and the polarizer/analyzer angle is indicated in the upper left corner of each photograph. The photographs of each sample were taken with the coating direction (movement direction of the sample stage) parallel to the polarizer in the photo on the left, and the coating direction at 45° with the polarizer in the photo on the right. On one hand, in the case of the LC coating without UV irradiation, planar LC alignment was recognized as shown in Fig. 4 (a). Previously, it has been repeatedly reported that planar alignment is maintained even when UV is irradiated with LC coating simultaneously [4]–[8]. On the other hand, in the case of the post-coating UV irradiation, vertical LC alignment was recognized as shown in Fig. 4 (b). The same observation was performed with a different host NLC, and the same alignment transition from the planar alignment to vertical alignment was confirmed. No such alignment transition was observed in the NLC to which UCL was not doped, and these are consistent with previous results. From these observations, there is no doubt that the alignment transition from the planar alignment to

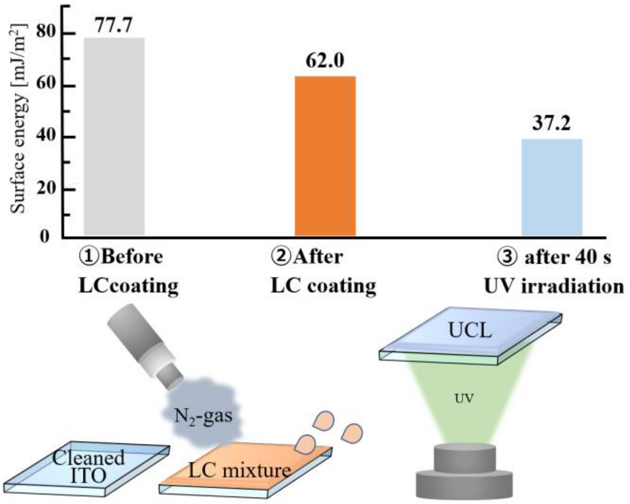


Fig. 5 Surface energy measurement. ① before LC mixture coating, ② after LC mixture coating, and ③ after 40 s UV irradiation, respectively.

vertical alignment occurs by post-UV irradiation.

It is interesting to know the mechanism by which the alignment transition occurs when added UCL is irradiated with UV light. We investigated whether or not UV polymerized UCL film is segregated locally at the substrate surface from the viewpoint of the surface energy change. Figure 5 shows the surface energy at each process by the contact angle method. Right after the substrates were cleaned by the plasma asher, the measured surface energy was 77.7 mJ/m^2. Then, the mixture of NTN-02 and UCL was once coated by slit coater onto these substrates, and they were divided into two groups. For the first group, the coated material on the substrate was removed by spraying high-pressure nitrogen gas, and then the surface energy was measured to be 62.0 mJ/m^2. This decrease in surface energy suggests that the mixture of NLC and UCL could not be removed by the high-pressure nitrogen gas incompletely and remains on the substrate surface. According to the previous photograph (Fig. 4 (a)), it seems that the surface is not in a state that would induce vertical alignment. For the second group, after UV irradiation for 40 seconds, the coated material on the substrate was removed by spraying high-pressure nitrogen gas. Next, the surface energy was measured and found to be 37.2 mJ/m^2. As is evident from the previous photograph (Fig. 4 (b)), it is reasonable to assume that UV irradiation lowers surface energy and induces vertical alignment. In other words, the substrate surface was covered by a UV polymerized mixture segregated on the substrate surface, which caused the appearance of vertical LC alignment.

Here, an idea came from Fig. 2 (a) because the right side of this molecular structure is similar to MMA. If vertical alignment could be achieved simply by lowering the surface energy through UV polymerization, inexpensive MMA could be used instead of UCL. In the next attempt, we spin-coated only MMA on the substrate and UV-polymerized it to form a thin film, whose thickness is approximately several hundred microns. When the surface energy for the UV polymerization MMA film was measured by the contact angle method, it was found to be 59.69 mJ/m^2, which was low enough to induce vertical alignment. However, as shown in Fig. 6, when NTN-02 was coated on the UV polymerized film surface, random planar alignment was obtained. These results suggest that the occurrence of vertical alignment essentially requires the involvement of the entire UCL.

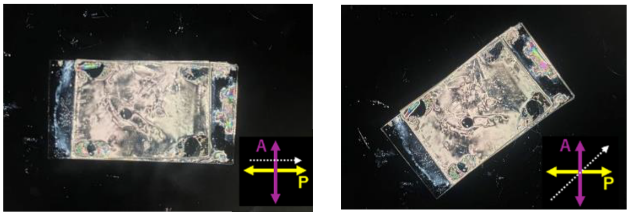


Fig. 6 Photographs of the alignment texture of coated NLC on the UV polymerized MMA.

Based on a series of studies, it is also necessary to consider the occurrence of mesoscopic-sized protrusion of the outermost surface of the UV-polymerized UCL layer. Figure 7 represents the AFM images at each process by the conventional dynamic mode scan in the atmospheric environment. Right after the substrates were cleaned by the plasma asher, the image observed with AFM showed almost flat, which is typical of an ITO electrode surface. Then, the mixture of NTN-02 and UCL was once coated by slit coater onto these substrates, and they were divided into three groups. For the first group, the coated material on the substrate was removed by spraying high-pressure nitrogen gas, and then the image observed with AFM also showed almost flat. In the previous experiment, the decrease in surface energy was small, therefore this is probably due to the fairly thin coated layer formed on the ITO electrode surface. For the second group, after UV irradiation for 40 seconds, the coated material on the substrate was removed by spraying high-pressure nitrogen gas, and then the AFM image was observed. As a result, no sharp protrusions such as in another article [15] were observed while a tiny surface unevenness remained. It is not surprising that the outermost surface of the polymerized UCL layer showed many cracks similar to those seen on the ITO electrode surface. This is because the polymerized UCL filling the grooves of the ITO electrode surface reflects simply the trace of the grooves on the ITO electrode surface in the order of tens of nanometers. There was another concern that the surface topology may be destroyed by spraying high-pressure nitrogen gas. Because it requires removing the outermost LC for AFM in the atmospheric environment. In-liquid (actually in-liquid crystal) AFM eliminates these concerns. Figure 8 represents the in-liquid AFM images at each process by the dynamic mode scan. Since the subject sample was not identical to that in Fig. 7, a conventional dynamic mode scan AFM image in the atmospheric environment was observed first, as shown in Fig. 8 (a). Next, after coating the NTN-02, the cantilever chip was immersed into the layer of NTN-02 and imaged with AFM, as shown in Fig. 8 (b). Here the magnification in Fig. 8 (a) and (b)

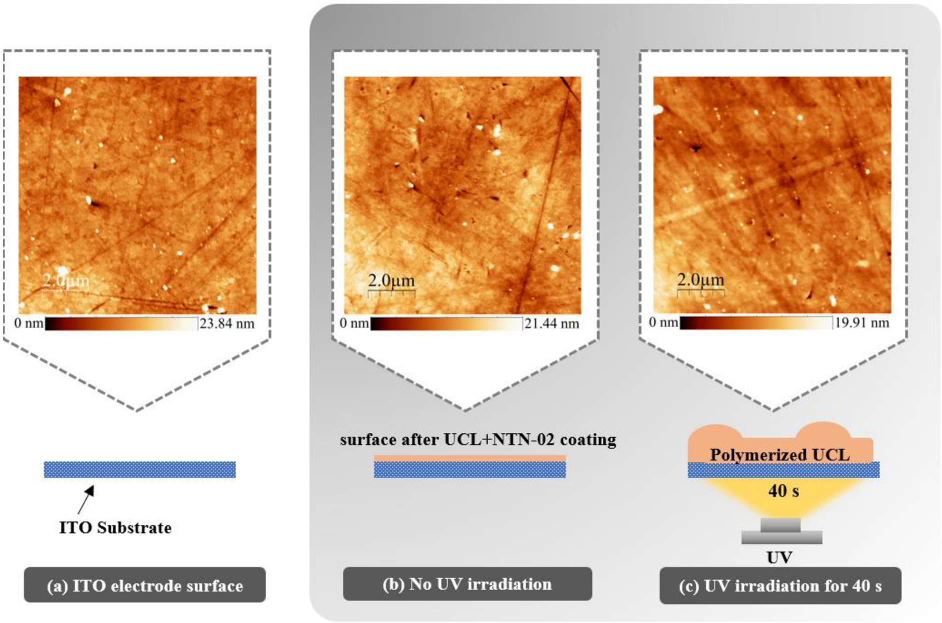


Fig. 7 AFM images at each coating and UV irradiating process by the conventional dynamic mode scan.

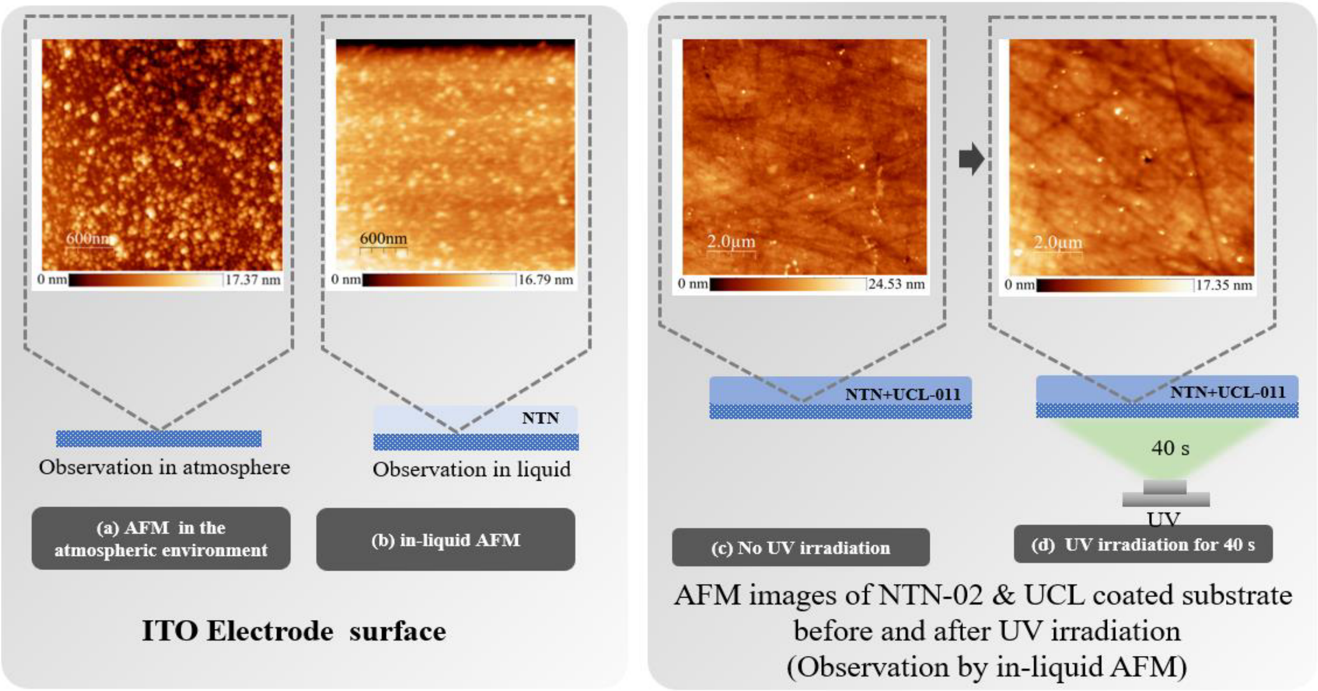


Fig. 8 In-liquid AFM images at each coating and UV irradiating process.

is higher than in Fig. 7, and a typical sputtered ITO grain pattern that were suspected to be caused by extraneous objects [24], [25] was observed. The ITO surface was almost flat, and less difference between Fig. 8 (a) and (b) was confirmed. This means that the LC molecules near the surface do not interfere with the movement of the cantilever tip. Figure 8 (c) shows in-liquid AFM images, where the cantilever chip was immersed into the layer of the mixture of NTN-02 and UCL after the slit coating process whereas UV light was not irradiated. It is found that the granular pattern of ITO has disappeared, and less unevenness can be observed on the surface. Finally, UV light was irradiated for 40 seconds then an in-liquid AFM image was obtained, as shown in Fig. 8 (d). It is found from this result that sharp protrusions do not occur even when UV polymerization is performed. In other words, it was confirmed that the hypothesis that the cause of vertical alignment transition is the occurrence of protrusions due to UV irradiation is incorrect.

A series of experimental results imply that the driving force of the LC alignment transition from the planar to vertical alignment induced by UV irradiation after slit coating is that the mesogenic group of UCL realigned vertically, as illustrated in Fig. 2 (c). The reason why the planar alignment is maintained when UV light is irradiated at the same time as the LC is coated by the slit coater is probably that the planar alignment regulation force generated by shear flow is fairly strong [26]–[28]. Let me consider the controllability of the pretilt angle by the slit coater method. It is interpreted that the reason for the poor reproducibility of the pretilt angle dependence on the UV irradiation condition [7] is that the pretilt angle was governed by an unstable torque balance inside the liquid crystal layer. Therefore, the mesogenic groups are forced to realign vertically when the shear flow disappears and UV polymerization proceeds. The mechanism by which vertical alignment is induced is thought to be similar to that using the photoisomerization reaction invented by Ichimura et al. as named 'command surface' [29], [30]. In our experiments, it seems that UV irradiation causes the realignment of mesogenic groups simultaneously with UV polymerization, resulting in an effect similar to that of photoisomerization reactions. Controlling the arbitrary pretilt angles may be difficult without developing materials that develop an optimal pretilt angle after polymerization [31]. Above all, the slit coater method itself is still in progress.

5. Conclusion

The role of UV-curable mesogen was studied to elucidate the leading cause of the LC alignment transition from planar to vertical alignment. AFM observations revealed that the UV-curable mesogen polymerized after processing by the slit coater realigned LC molecules solely through intermolecular interactions without any change in surface topology. The tilt angle of the mesogenic groups seems not to develop a gradual change by UV irradiation. Novel methods for controlling the pretilt angle and suitable materials will also be invented when a superior instrument capable of observing intermolecular interaction directly is developed.

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