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Atomic force microscopy of mechanically rubbed and optically buffed polyimide films

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Polymer coated substrates modified both by mechanical rubbing and optical buffing have been found to cause liquid crystal molecules to align. Atomic force microscopy (AFM) was used to characterize the differences and similarities in polyimide substrate coatings after being subjected to these two processes. Though the buffing processes caused similar alignment on the surfaces, it was found that the mechanical rubbing created grooves on the order of 250 nm, whereas optical buffing resulted in no changes to topographic structure on the order of 100 nm scale and some variations at smaller scales. From this observation it was confirmed that the interaction causing the alignment must be associated with molecular alignment rather than large scale physical grooving.

1. Introduction

With the increasing use of liquid crystal displays, there is strong motivation to elucidate the mechanisms by which the manipulation of liquid crystal molecules (LCMs) can be achieved. LCM alignment has been achieved through interactions with the supporting substrates [1] which are useful in producing reproducible alignment [2]. These substrates are prepared by mechanical rubbing or by exposure to polarized light causing a photochemical *trans-cis/cis-trans* isomerization [3-6]. Mechanical preparation of the substrates involves rubbing the surfaces with a lens tissue, cloth, etc., producing grooves of dimensions comparable to the grit of the rubbing material [2, 7, 8]. Berreman [1] determined that LCMs orient parallel to, rather than across, the grooves or ridges to minimize the elastic strain energy. Mechanical rubbing is performed on both bare substrates and on those coated with polymer films (aligning agents), though the latter has become more widely used [8]. The second method of preparation is through the use of optical aligning agents. Comparison of the effectiveness of optical buffing and mechanical rubbing has resulted in debate over the size scale of interaction responsible for alignment [7, 9, 10]. One view is that since rubbing produces grooves in the polymer coating as well as the substrate, a physical interaction with the grooves produces the alignment [1, 9]. Gibbons *et al.*

[11] and Shannon *et al.* [12] have shown that LCM alignment can also be achieved by exposing spin-coated polymer films to polarized light. There is compelling evidence that both in the case of rubbing and of optical alignment there is a molecular reorientation of the polymer chains that causes alignment [2, 7-9, 14-18]. It remains unclear, however, whether the molecular interaction is the necessary and sufficient condition, i.e. whether nanoscale grooves are also contributing to alignment.

In the present study we compare mechanically rubbed and optically buffed polymer coatings, with and without dye, using AFM. Analyses of surfaces modified by mechanical rubbing and exposure to polarized light provides a framework in which to compare the effects of molecular orientation, assumed to occur in both cases, with topographic effects. To our knowledge this is the first study performed to compare simultaneously the surface structure of polymer coatings modified by these two processes.

2. Experimental procedure

In order to compare the effects of surface modification on topographic structure, AFM was performed on six samples which included polymer films, with and without dye, in three states of modification: unaltered, mechanically rubbed, and optically buffed. The glass substrates were spin-coated with a thin layer of 0.5 wt % of a silicone polyimide copolymer in NMP solvent. For

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samples of dyed polymer films, the polyimide was doped with a diazodiamine dye at a dye:polyimide ratio of 1:2 by weight. The chemical structure and absorption spectrum of the dye are described in [11]. The samples were then cured and mechanically rubbed or optically aligned.

The topographic analysis was carried out on a Dimension 3000 AFM† in intermittent contact mode to minimize deformation on the polymer. Scan sizes from 1–50 μm were obtained using silicon cantilevers oscillating near the resonant frequency of 320–440 kHz and scanned at a frequency of 0.5 Hz. Images are presented without filtering, with the exception of a linear background subtraction.

3. Results and discussion

Comparison of the surface structures resulting from different modification processes on dyed and undyed polymer films exhibited dramatic differences. Figures 1(a) and 1(b) (5 μm^2) show the topographic structures of unaltered polymer films with and without dye. In the film without dye, it is apparent that the spin-coated polymer contains clusters approximately 150–200 nm in diameter with heights on the order of 10 nm. The clusters are separated by about the same distance as they are wide, resulting in a relatively smooth undulating surface. The surface of the dyed polymer, on the other hand, contains large clusters with comparable widths and heights of an order of magnitude larger than that of the undyed polymer. The clusters on this film are around 200 nm high and spaced as much as 1 μm apart. In smaller scans, such as the 1 μm scans in figures 2(a) and 3(a), it is possible to see that the surfaces *between* clusters of the dyed and undyed polymer films appear quite similar; thus, the main difference between the two films is the formation of large clusters in the one containing dye.

The effects of modification on the two types of films will be discussed separately. Figure 2 is a matrix of images showing the topological result of surface modification on the undyed polymer at different size scales. From left to right, the columns distinguish the unaltered, mechanically rubbed, and optically buffed samples, whereas the rows show the structure at increasing size scales from top to bottom (1, 5 and 25 μm^2). Starting from the smallest scan size, figures 2(a), 2(b) and 2(c) show that mechanical rubbing tends to pepper the smooth undulating surface with smaller particulates, whereas optical buffing preserves the scale of the undulation,

† Digital Instruments Dimension 3000: experiment performed in the Scanning Probe Imaging Facility at the Laboratory for Research on the Structure of Matter at the University of Pennsylvania.

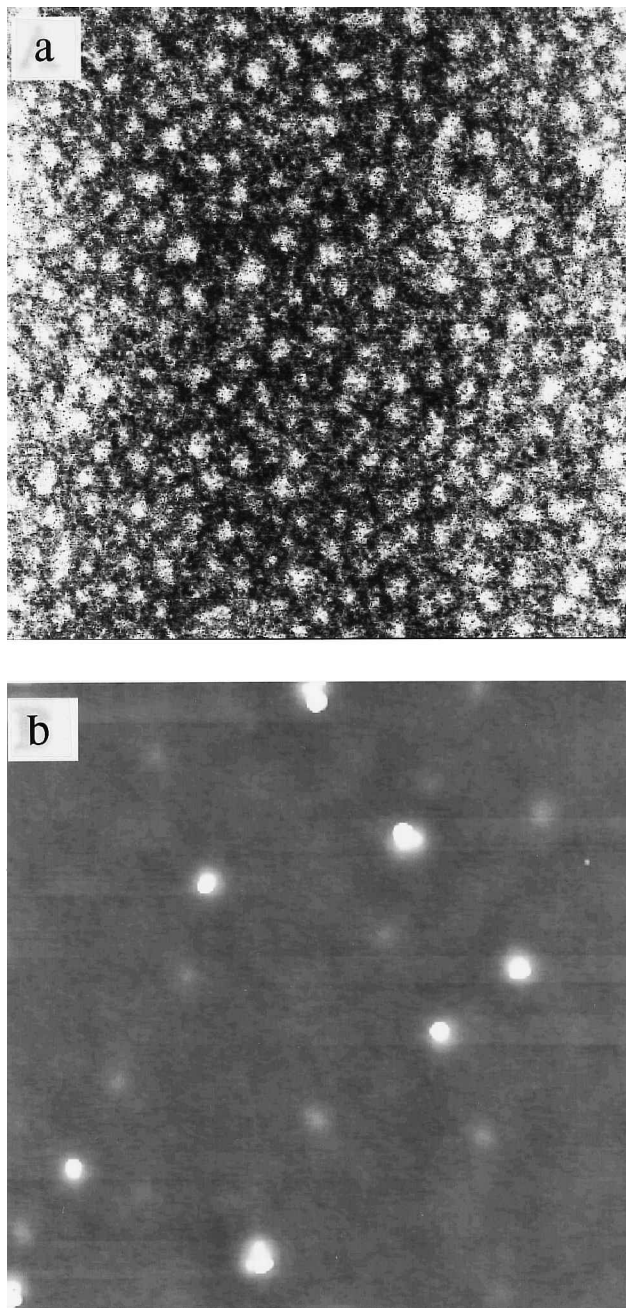


Figure 1. Unaltered polymer film (a) without dye, (b) with dye.

increasing the vertical height only by a factor of two. At a larger scale, figures 2(d), 2(e), and 2(f), the ridges from the mechanical rubbing become apparent. At this size scale it is easier to see the tendency for the clusters to aggregate. Finally at the largest size scale it is easier to see the tendency for the clusters to aggregate. Finally at the largest size scale, figures 2(g), 2(h), and 2(i), the effects of mechanical rubbing appear to be dramatic. While the optically buffed sample shows little difference

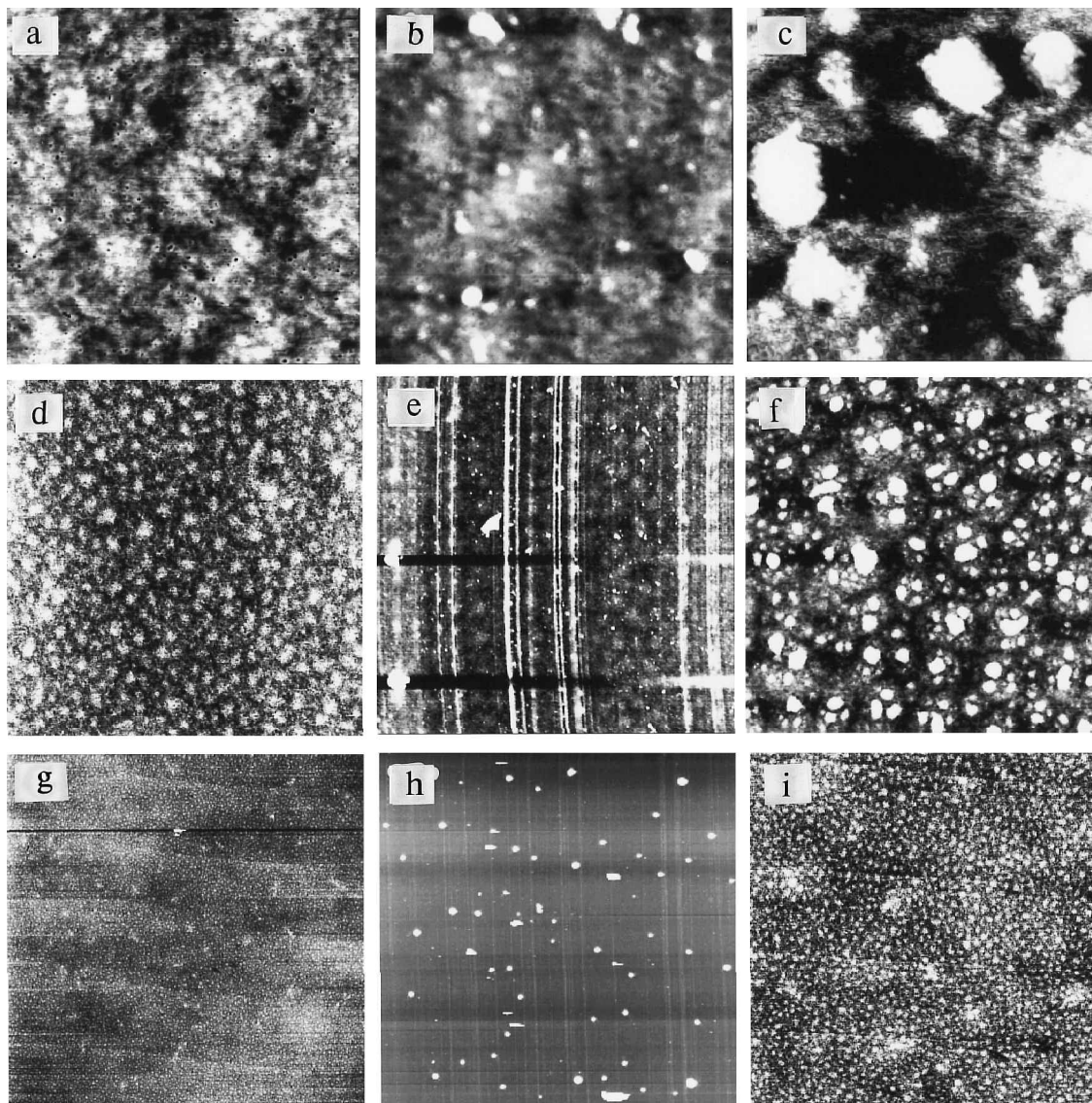


Figure 2. Modification of polymer films, without dye.

from the unaltered sample, other than the aggregation of previously evenly spaced clusters, the mechanically rubbed sample contains clusters that are much larger. While the lateral dimensions of the clusters are similar to those observed in unaltered and optically buffed samples, the height is greater, 250 nm. In addition, the spacing of the clusters is $5\mu\text{m}$, which explains the absence of such large clusters in images at the previous size scale. The tendency for the clusters to be adjacent to the ridges is also noted.

Figure 3 shows the surface morphology of the dyed polymer in the same matrix as figure 2. Comparing the first and third columns, it is apparent that the optical buffing produces little change in the surface morphology. The size of the clusters formed as the result of the addition of dye makes it difficult to see whether a similar

aggregation of smaller clusters in the undyed films at the moderate size scale, as shown in figures 2(d) and 2(f), occurs in the dyed polymer coatings. The changes in surface structure due to mechanical rubbing, however, as shown in the second column, are observed to be dramatic. In addition to the formation of ridges, as expected from the process, the large clusters are no longer present. The lateral widths of the clusters present on the mechanically rubbed surface, pictured in figures 3(e) and 3(h), are similar to those present in the unaltered and optically buffed samples. The cluster heights, however, are significantly different as the height is only 15 nm in the mechanically rubbed sample as compared with the 250 nm cluster height in the unaltered and optically buffed samples. It is again observed that the clusters are adjacent to the ridges.

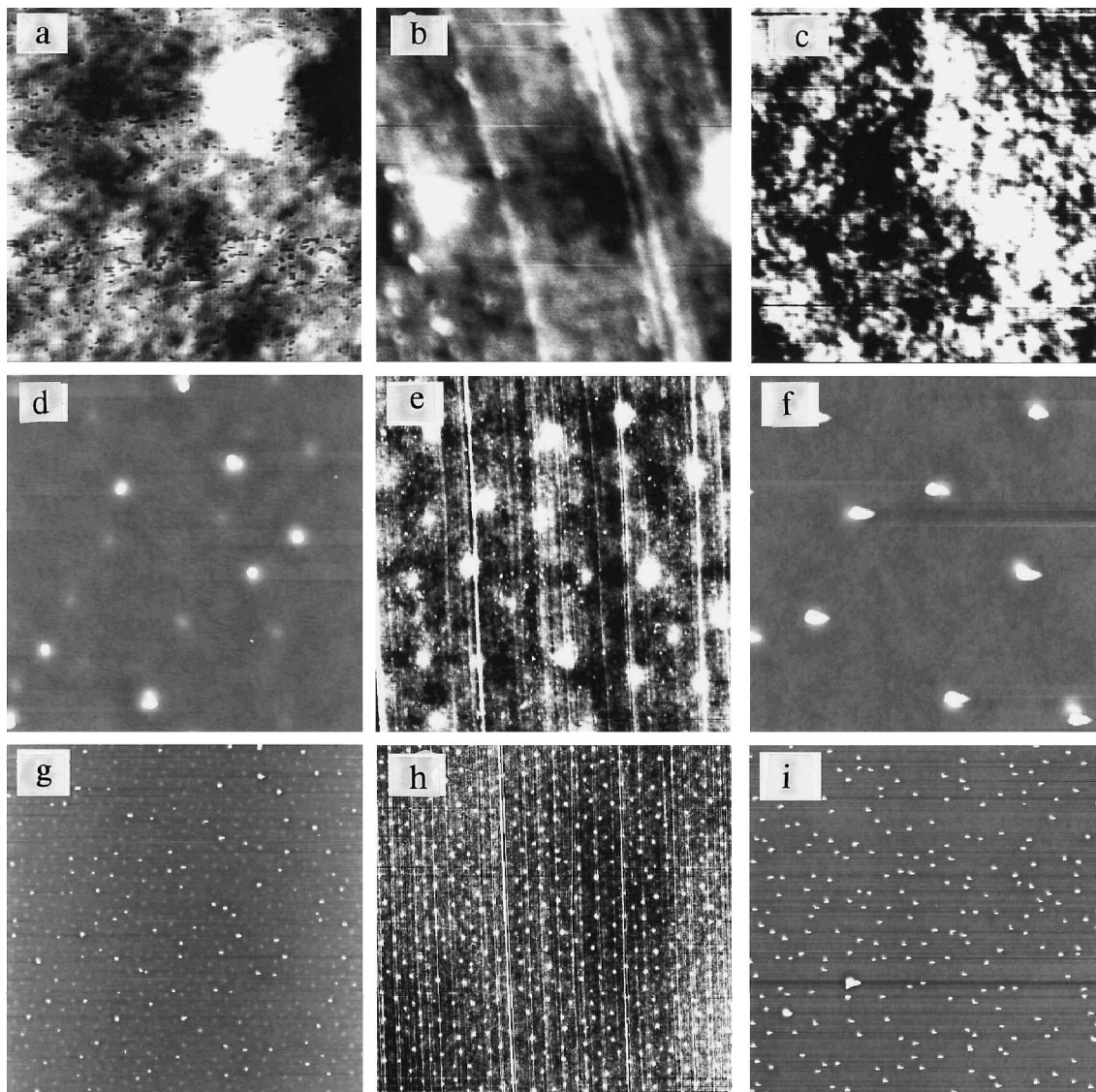


Figure 3. Modification of polymer films, with dye.

These measurements show that physical grooves or ridges are not present on the optically buffed films, at least to the scale of 10 nm, whereas grooves are obvious on mechanically rubbed films. At size scales on the order of $1\ \mu\text{m}$ and above, there is no observable topographic effect due to optical buffing. As noted above, on the nm scale there are topographic differences but these do not appear to be aligned in ridges. Since the surface structure of the mechanically rubbed and optically buffed samples differ on larger scales, but achieve similar alignment of LCMs, the size scale of interaction responsible for the LCM alignment is concluded to be smaller than $1\ \mu\text{m}$, i.e. the groove dimension.

4. Conclusions

Since the alignment of LCMs was observed on both optically buffed and mechanically rubbed films, despite

the absence of grooves in the former case, it is concluded that external modification need not create macroscopic features in order to align LCMs. In addition, since the alignment in both cases is similar, it follows that the size scale of interaction responsible for alignment should be the same. Therefore, it has been determined that, if any surface similarities are to be found between the two types of modified films, the size scale of interest is less than a 100 nm.

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