Alignment of liquid crystals on polyimide films exposed to ultraviolet light

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The influence of unpolarized and linearly polarized UV exposure on previously rubbed as well untreated thin polyimide (PI) alignment layers was studied. Optical retardation and surface morphology measurements were carried out to understand the nature of surface modification as a function of the polarization and the exposure time of the UV light under different surface conditions. The exposure of the UV light on the PI layer was found to change drastically the morphological anisotropy due to photochemical dissociation. The control of anisotropic surface forces by the linearly polarized UV (LPUV) exposure combined with the rubbing process is important to study the alignment mechanism of liquid crystals on various substrates. A simple model incorporating the effect of the LPUV exposure is presented together with the essential features of the experimental results. [S1063-651X(98)15005-5]

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I. INTRODUCTION

Substrates with anisotropic surface potential such as obliquely evaporated SiO layers, Langmuir-Blodgett films, and rubbed polymer films have been used to control the alignment of the optic axis of liquid crystals [1]. Mechanical rubbing of polyimide (PI) layer is the most common method used in mass production of liquid crystal (LC) displays because of its simplicity and thermal stability. Although the mechanism responsible for the resultant alignment is not yet fully understood, it is believed that shearing of the film during the rubbing process orients polymer aggregates or polymer chains along the rubbing direction, as revealed by previous surface studies [2-5] employing atomic force microscopy (AFM) and optical birefringence measurement. The disadvantages of the rubbing method are the generation of dust particles, electrostatic charges, physical damage, and nonuniformities which are detrimental to the fabrication of thin film transistor based devices.

To eliminate these problems, a nonrubbed photoalignment process has been developed. It has been demonstrated that poly(vinyl)4-methoxycinnamate and poly(vinyl)cinnamate films, when exposed to a linearly polarized ultraviolet (LPUV) light, can be very effective as alignment layers [6,7]. The photoalignment method allows for an easy control of the alignment direction and anchoring strength so that multidomain devices, with improved viewing angle characteristics, can be readily produced. However, this method yields alignment layers that possess poor thermal and chemical stability and reliability compared to the rubbing method.

Recently, several research groups have reported alignment of LC's by PI films exposed to the LPUV light [8,9]. Fourier transform infrared (FTIR) spectroscopy has shown that the UV irradiation anisotropically photodissociates photosensitive chemical bonds in PI's including those in the imide ring [10]. This reduces the polarizability of PI molecules [11] and, as we will show here, changes the surface morphology. Since the LC alignment is believed [12] to de-

pend on the competition of various physical and chemical interactions between the LC molecules and the substrate surface, it is very important to determine the role of surface morphology in LC alignment. To the best of our knowledge, there have been no such systematic studies of UV exposed PI layers.

In this paper, we report the results of the influence of LPUV light on surface properties of thin PI films. We have measured the optical birefringence (retardation) and characterized surface morphology with AFM for various UV exposures and surface conditions. We also present a phenomenological model to help understand the relationship between the measured phase retardation, surface morphology, and LC alignment.

II. EXPERIMENT

The PI used in this study is SE610 of Nissan Chemical Company. Spin coated films of SE610 were soft baked at 100 °C for 15 min, then hard baked at 220 °C for 1 h. The film was exposed to the LPUV light from a Xe lamp. The intensity of the UV light after passing through the polarizer is approximately 6 mW/cm². A metal cylinder wrapped with velvet, spun with a linear velocity of 1.1 m/min, was used to rub the films.

We used a photoelastic modulator (PEM90, Hinds Instruments) with a fused silica head and a He-Ne laser for optical phase retardation measurements. The photoelastic modulator (PEM) was placed between two crossed polarizers with its optic axis at 45° to the axes of polarizer and analyzer. The LC cell prepared with photoalignment layers was placed between the PEM and the analyzer. The signal from the photodetector was fed to a lock-in amplifier (EG & G Princeton Applied Research, Model 5210) for measuring the ac signal and a digital multimeter for the dc signal. The lock-in amplifier was tuned to the 50-kHz reference signal from the PEM. The laser beam was incident normal to the sample cell's surface. The signal was monitored while rotating the

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FIG. 1. Optical phase retardation as a function of the rotation angle of the sample for (curve a) unrubbed, (curve b) rubbed, and (curve c) LPUV exposed (30 min) PI films.

sample with respect to the surface normal. The sensitivity of this method enables us to measure the phase retardation with a precision of $\pm 0.01^{\circ}$.

Surface morphology was measured with a commercial atomic force microscope (Nanoscope III, Digital Instruments Inc.) operated in a contact mode with a constant force. The measurements were made in air at room temperature using a microfabricated pyramidal shaped Si_3N_4 tip integrated into a rectangular cantilever with a spring constant of 0.58 N/m.

III. RESULTS AND DISCUSSION

The optical phase retardation was measured as a function of the angle of rotation for an unrubbed PI film, a PI film exposed to LPUV for 30 min, and a rubbed film (see Fig. 1). The rubbing direction and the LPUV light's polarization axis were kept collinear but perpendicular to the PEM's optical axis. Under these conditions, the sample birefringence causes the optical phase retardation to vary at twice the rate of the rotation frequency of the sample. The sign of the retardation was determined from the phase difference. The fast and slow axes of the sample can be determined from the amplitude and phase of the optical retardation.

A. Alignment on unrubbed PI films exposed to LPUV light

An unrubbed PI film normally shows a negligible optical retardation, typically less than 0.05° , due to the flow-induced ordering of the PI chains during the film deposition process and/or the strain-induced birefringence of the glass substrate. The dashed curve (*a*) in Fig. 1 represents the optical retardation of such unrubbed sample.

The optical retardation of a PI film increases drastically upon rubbing, as evident from curve (b) in Fig. 1. It is believed [2,3] that the alignment of polymer chains caused by rubbing is the origin of such a large optical anisotropy. Optical retardation of PI films also exhibits rapid increase upon LPUV exposure. The result of the film exposed for 30 min is represented by the dotted curve (c) in Fig. 1. Clearly, the magnitude of the retardation is comparable, but has an oppo-



FIG. 2. Optical phase retardation as a function of the LPUV exposure time for an unrubbed PI film. The solid line represents the fit of the data to Eq. (2).

site sign to that of the rubbed film. This indicates that the LPUV exposure causes anisotropic photochemical dissociation of imide bonds parallel to its polarization, leaving the polymer chains in the perpendicular direction relatively unperturbed. This is consistent with the conclusion drawn by a previous FTIR study [10]. The broken bonds reduce the polarizability of the PI molecules. In contrast to the previous report that the LC alignment is mainly achieved via the interactions of LC molecules with the polar functional group in PI produced by LPUV light [13], our results show that anisotropic irreversible depolymerization is primarily responsible for LC alignment on SE610 PI films exposed to LPUV light.

Within the framework of the previous model [11], the photoreaction rate can be described by a coupling term $\vec{m} \cdot \vec{p}$, where \vec{m} and \vec{p} are the transition moment of the photosensitive bonds and the polarization direction of LPUV light, respectively. The time dependent angular distribution of photosensitive bonds can be written as

$$N(\theta, \phi, t) = N_0(\theta, \phi) \exp(-\alpha t \cos^2 \phi \cos^2 \theta), \qquad (1)$$

where $N_0(\theta, \phi)$ is the initial angular distribution of photosensitive bonds, and α is a constant. Here ϕ and θ denote the azimuthal and polar angles of \vec{m} with respect to \vec{p} , respectively. The initial distribution of photosensitive bonds without any surface treatment is assumed to be azimuthally isotropic, i.e., $N_0(\theta, \phi) \rightarrow N_0(\theta)$. Considering a twodimensional system in which all molecules lie in a plane parallel to the substrate, the total optical retardation of such a film depolymerized by the LPUV light can be written as

$$R(t) = A \int_{0}^{\pi/2} (2\cos^{2}\phi - 1) \times \{1 - \exp(-\alpha t \cos^{2}\phi \cos^{2}\theta)\} d\phi, \qquad (2)$$

where A is a constant. The optical phase retardation as a function of the UV exposure time is shown in Fig. 2. The optical anisotropy rapidly increases with the exposure time, and becomes saturated in 1 h, after which it begins to de-



FIG. 3. The time evolution of polymer chain alignment with increasing the LPUV exposure time. The arrows indicate the increase in the exposure time. Rods represent molecular units of the polymer.

crease slowly. This may be due to the fact that the prolonged UV exposure eventually dissociates even the bonds oriented perpendicularly to the polarization direction. The solid line in Fig. 2 represents the fit of the data to Eq. (2) with α =0.026. Assume that the spin coated PI film can be treated as a two-dimensional system composed of interconnected rods forming the polymer chains. If the absorbed photon energy exceeds the photosensitive bond energy, the polymer chains are photochemically dissociated (i.e., the link between the rods is broken). In Fig. 3, the time evolution of the polymer chain density and orientation is shown during LPUV exposure. The polymer chains are modeled as consisting of 1000 rods. Initially, the rods are distributed isotropically. With increasing LPUV exposure time, an increasing number of polymer chains, oriented with their constituting rods parallel to the polarization direction, is dissociated, thus leaving intact those chains which are oriented perpendicularly. Since the majority of remaining polymer chains are aligned nearly perpendicular to the LPUV light's polarization, the stacks of polymer segments are observed by AFM as macroscopic domains elongated in that direction.

Since the LC alignment occurs at the substrate surface, it is very important to understand the role of surface morphology in LC alignment. Figure 4 shows the surface morphologies of PI films determined by AFM for different LPUV exposure times. The unexposed PI film shown in (a) consists of randomly distributed circular domains with diameter of 100–200 nm and root-mean-square (rms) vertical roughness of 0.4 nm. After 1 h of LPUV exposure, as shown in Fig. 4(b), elongated polymer aggregates 100–200 nm long and 50–100 nm wide, were formed in the direction perpendicular to the LPUV polarization. The roughness of the surface becomes 0.2 nm. The resultant two-dimensional power spectrum shown in Fig. 4(c), obtained by Fourier analysis of Fig. 4(b), clearly reveals a new component in the direction per-



FIG. 4. Surface morphologies of the unrubbed PI film determined by AFM for various LPUV exposure times: (a) 0 h, (b) 1 h, (d) 2 h, and (e) 4 h. The two-dimensional power spectrum of (b) obtained by a fast Fourier transformation is shown in (c).

pendicular to the polarization of UV light (shown by the arrow). The degree of anisotropy is smaller than in the rubbed film. The anisotropy of surface morphology has also been confirmed by x-ray reflectivity experiments [14]. We believe that the formation of polymer aggregates caused by the photochemical dissociation and associated morphological anisotropy plays a very important role in LC alignment. With increasing exposure time, the surface roughness also increases to 0.28 and 0.38 nm for 2- and 4-h [Figs. 4(d) and 4(e)] exposures, respectively. In all cases, the polymer aggregates are preferentially oriented perpendicular to the polarization direction of the UV light.

To understand the effect of surface morphology on LC alignment, we observe a microscopic texture with a nematic LC, Merck-E48, in a 6- μ m-thick cell. The best alignment was obtained with 30–60 min of UV exposure of both surfaces. The alignment began to degrade with longer exposure. Sample cells with 4-h exposure exhibited microscopic domains, and were less uniform than cells with 30-min exposure. This may depend on two factors. One is the possibility of reverse tilt at the surface. Since we exposed LPUV light normal to the surface, it is possible to create pretilt angles in two opposite directions with equal probability. The other is the effect of surface roughness. On the basis of AFM results, it was found that the degree of alignment decreases with



FIG. 5. The LC alignment texture (a) and surface morphology (b) for a rubbed PI film; (c) and (d) show the texture and surface morphology, respectively, of the rubbed sample subsequently exposed to RPUV light.

increasing surface roughness. This is consistent with the fact that the surface ordering decreases with increasing isotropic surface roughness [15]. However, it should be pointed out that the roughness anisotropy, which appears to govern the LC alignment, cannot be reliably measured from AFM images.

B. Alignment on rubbed PI film exposed to unpolarized UV light

Since the azimuthal angle of the easy axis of a LC is strongly influenced by the distribution of photosensitive bonds [11], it is important to determine the dependence of the anisotropic surface interactions, induced by LPUV light, on the initial distribution of the photosensitive chemical bonds produced by rubbing.

Figure 5 shows microscopic LC texture in a $6-\mu$ m-thick cell and surface morphology of the rubbed PI film before [(a)and (b)] and after [(c) and (d)] its exposure to randomly polarized UV (RPUV) light. Very uniform LC alignment was achieved before UV exposure. Surface morphology of the rubbed surface clearly shows microscratches of different widths and depths, and elongated polymer clusters along the rubbing direction. The alignment and hence the texture of this cell changed drastically after 15-min exposure to RPUV light, as shown in Fig. 5(c). This kind of the Schlieren texture is usually obtained for unrubbed substrates. Since the polymer chains in the unrubbed sample aligned randomly (and hence isotropically), it may be concluded that the RPUV light dissociates the polymer chain in all directions rendering it isotropic. The properties of the RPUV exposed surface are expected to be similar to an unrubbed substrate. As is clear from Fig. 5(d), many of the scratch lines, especially finer ones, become obscured to differing degrees. However, the large scratch lines still remain. It appears that microscopic scratches, at a length scale smaller than a critical dimension, determine LC alignment in much the same way



FIG. 6. A homogeneously aligned cell between crossed polarizers. The cell rubbing direction R coincides with the axis of one of the crossed polarizers. Dark (bright) regions marked as I (II) are without (with) LPUV exposure.

as relief gratings [16]. This suggests that macroscopic scratches (mechanical grooves) which are parallel to the rubbing direction are inconsequential for the LC alignment. This conclusion is consistent with the inferences drawn on the basis of x-ray reflectivity measurements of surface morphological anisotropy as those are over a length scale of x-ray coherence length ($< 0.5 \ \mu$ m) [14].

C. Alignment on rubbed PI film exposed to LPUV light

When a rubbed PI film is exposed to LPUV light, the competition between the effects of rubbing and LPUV light determines the direction and the degree of alignment [17]. Figure 6 shows the microscopic textures of a sample that was rubbed twice and then exposed to LPUV light for 20 min. The polarization direction of LPUV (marked as "UV" in Fig. 6) light makes an angle of 40° with respect to the rubbing direction. Only half of the surface is exposed to LPUV light, so that the exposed and unexposed areas could be compared. The rubbing direction R coincides with the axis of one of the polarizers, and minimum transmittance is obtained in the unexposed region (marked as I), as expected. In the exposed region (region II), a dark state can be obtained by rotating the LC cell by 50° with respect to the axis of the same polarizer. To further understand the effect of LPUV light, we determine the changes in surface morphology with AFM. Prior to LPUV exposure, microscratches and PI clusters extending in the direction of rubbing are clearly visible as shown in Fig. 5(b). After LPUV exposure, the prominence of these scratches and hence the anisotropy was diminished as evident from Fig. 7. Interestingly, the PI microclusters now appear to be elongated in the direction perpendicular to the polarization. It is believed that the photoreaction process is responsible for their formation. The power spectrum shows a new branch at an angle of $\sim 50^{\circ}$ with respect to the rubbing direction. This subtle change in surface morphology profoundly changes the LC alignment direction, as shown in Fig. 6. Since the easy axis of LC alignment coincides with the fast optic axis of the PI films, the angle $\Delta \xi$, which represents the deviation of the alignment direction from the direction normal to the polarization of LPUV, can be determined from the measured angular dependence of film birefringence. Figure 8 shows the angular dependence of the



FIG. 7. Surface morphologies obtained by AFM and its power spectra for the rubbed PI film with LPUV exposure at an angle of 40° .

film birefringence in polar coordinates for the twice rubbed PI film with LPUV exposures of 0 min (open circles), 5 min (filled squares), and 20 min (open triangles). After approximately 20 min, the birefringence and LC alignment direction is effectively rotated by 50°. The dependence of $\Delta \xi$ on the exposure time is shown in Fig. 9 for films rubbed two and four times. Clearly, with increasing exposure time, $\Delta \xi \rightarrow 0$. It should be possible to utilize these observations to control and fine tune the LC alignment on a rubbed PI surface by adjusting the LPUV exposure time.

To understand the dependence of $\Delta \xi$ upon LPUV exposure, one needs to consider the initial distribution of polymer chains and photosensitive bonds in the rubbed film. The orientational distribution that polymer chains acquire during rubbing can be assumed to be a Gaussian peaked in the direction of rubbing. However, LPUV causes a selective dissociation of the C(O)=N bonds, which are nearly parallel to the polymer chain. Assuming that the azimuthal and polar distributions are independent of each other, Eq. (1) can be modified as



FIG. 8. The angular dependence of the optical anisotropy for the twice rubbed PI film subsequently exposed to LPUV light with a polarization at 40° to the rubbing direction. The open circles, filled squares, and open triangles represent exposures for 0, 5, and 20 min, respectively.



FIG. 9. Dependence of $\Delta \xi$ on the LPUV exposure time. The circles and triangles denote the experimental data for the PI films rubbed two and four times, respectively. The solid lines represent the best fits of the data to Eq. (4). The dashed and dotted lines are the calculated curves for A = 0.1 and 0.5 min/deg, respectively.

$$N(\theta, \phi, t) = N_0(\theta) \exp\left[-\frac{1}{2} \left(\frac{\phi - \phi_b}{w}\right)^2\right] \\ \times \exp\left[-\alpha t \,\cos^2(\phi_0 - \phi)\cos^2\theta\right], \quad (3)$$

w being the width of the distribution. Here ϕ_b and ϕ_0 denote the azimuthal angles for the rubbing and LPUV directions, respectively.

Figure 10 shows the distribution of photosensitive bonds according to Eq. (3) for different LPUV exposure times and for $\alpha = 0.026$, w = 5, $\theta = 0$, and $\phi_b = 0^\circ$. Before UV exposure, the polymer chains possess a Gaussian distribution acquired during rubbing [Fig. 10(a)]. When it is exposed to



FIG. 10. Orientational distribution of photosensitive bonds calculated from Eq. (3) for different LPUV exposures on the rubbed PI film. (a) is the distribution with no exposure; (b) and (c) are the distributions after 10 min and 30 min exposures, respectively, with $\phi_0 = 40^\circ$; and (d) and (e) are the distributions after 30- and 60-min exposures, respectively, with $\phi_0 = 0^\circ$. For all curves, $\alpha = 0.026$, w = 5, $\theta = 0^\circ$, and $\phi_b = 0^\circ$.



FIG. 11. Optical phase retardation and phase difference as a function of the LPUV exposure time for the rubbed PI film for which polarization and rubbing directions were parallel.

LPUV light with polarization oriented at 40° with respect to the rubbing direction, the peak shifts in the direction perpendicular to the polarization of LPUV light, and the distribution height decreases with increasing the exposure time [Figs. 10(b) and 10(c)]. However, the optical phase retardation (Fig. 8) shows different behavior, i.e., the maximum value first decreases and then increases with increasing the exposure time. This appears to be due to photodissociation of bonds in the bulk of the film. Let $\phi_b = 0$ in Eq. (3); then the LC alignment determined by the LPUV exposure ϕ_s satisfies the equation

$$t \sin 2(\phi_0 - \phi_s) + A \phi_s = 0,$$
 (4)

where $A = (1/w)/(2\alpha \cos^2\theta)$. The solid lines in Fig. 9 represent fits of the experimental data to Eq. (3) for $\phi_0 = 40^\circ$ and $\Delta \xi = \phi_s + 50^\circ$. From these fits, we obtain $A = 0.24 \pm 0.01$ and 0.36 ± 0.01 min/deg for films rubbed two and four times, respectively. If the exposure time t is comparable to the value of A, then the angle of rotation lies between 0° and 50° , and the magnitude of birefringence is lower than its initial value. With increasing LPUV exposure, the easy axis rotates towards the alignment direction "preferred" by LPUV light, i.e., $\Delta \xi \rightarrow 0$. The width of the Gaussian distribution, w, is related to the surface anchoring energy in such a way that w decreases with increasing rubbing strength. Therefore, it will take longer UV exposures to rotate the easy axis. The dashed and dotted lines in Fig. 9 are the numerically calculated results for A = 0.5 and 0.1 min/deg, respectively. These results suggest that it should be possible to fine tune the alignment direction of the LC molecules by adjusting the LPUV exposure and/or the rubbing strength. Understanding the depen-



FIG. 12. The angular dependence of optical anisotropy for the twice rubbed PI film for different LPUV exposure times with $\phi_0 = \phi_b = 0$. The filled circles, open circles, open squares, and filled squares represent exposures for 0, 10, 30, and 60 min, respectively.

dence of α and w on the microscopic properties of LC and polymer material parameters is crucial for the effective use of this method.

Figure 11 shows the measured optical anisotropy and the phase difference as a function of the exposure time when the polarization direction of LPUV light coincides with the rubbing direction (i.e., $\phi_0 = 0$). The optical anisotropy created by rubbing rapidly decreases and vanishes after approximately 20 min. The phase angle changes abruptly from 0° to 180° at that time. For longer exposures, the anisotropy dramatically recovers and saturates within 1 h. The angular dependence of optical anisotropy on the exposure time are shown in Fig. 12. Interestingly, each branch points in the same direction, which is in contrast to the results shown in Fig. 8. However, from the phase change, we note that the branches are discontinuously rotated by 90° for exposure of 30 and 60 min. In the case of Fig. 8, the directions of R and UV light were different. Both results show that the populations of polymer chains parallel and perpendicular to the rubbing direction change abruptly due to chemical dissociation. Eventually, more chains remain intact in the direction perpendicular to the direction of polarization. Figures 10(d) and 10(e) show distributions of photosensitive bonds calculated using Eq. (3) for different exposures with $\phi_h = \phi_0 = 0$. It is very interesting that there are two symmetric peaks at $\phi = \phi_s$ and $-\phi_s$, different from the asymmetric peak in case of $\phi_0 = 40^\circ$ [Figs. 10(b) and 10(c)]. This suggests that there are two easy axes. These peaks move toward $\phi = \pm 90^{\circ}$, which becomes the final alignment direction dictated by LPUV exposure. For intermediate exposures, the two easy axes compete with each other, and the resultant axis does not rotate (i.e., $\phi_s^{av}=0$) for $-45^\circ < \phi_s < 45^\circ$ because polymer chains are distributed with equal probability in both directions. However, ϕ_s^{av} abruptly becomes $\pm 90^\circ$ for $\phi_s > 45^\circ$ or $\phi_s < -45^\circ$. At $\phi_s = \pm 45^\circ$, since both component (i.e., parallel and perpendicular to the rubbing direction) have equal probabilities, the average optical anisotropy is reduced to zero. The discussion and the model presented here provides a satisfactory explanation of the measured optical retardation.



FIG. 13. Surface morphologies of the rubbed and subsequently LPUV exposed PI films determined by AFM for different UV exposure times with $\phi_0 = \phi_b = 0$; (a) 0 h, (b) 1 h, (c) 4 h, and (d) 8 h.

Figure 13 shows surface morphologies of the rubbed samples exposed to LPUV with polarization parallel to the rubbing direction for different exposure times. Without UV exposure, scratch lines caused by rubbing are clearly defined, and LC's are aligned along the rubbing direction [Fig. 13(a)]. The visible lines are about 1 nm deep and 50–200 nm wide. With increasing exposure, the surface structure gradually become obscure. Though the wider of the scratch lines remain qualitatively the same, their depth decreases. For exposures of 1 h, the depth is reduced to 0.7–0.8 nm [Fig. 13(b)]. For

samples exposed for 4 and 8 h, the depth is only 60% and 30% of the original value, respectively [Figs. 13(c) and 13(d)]. It indicates that the surface anisotropy induced by the rubbing process is diminished by LPUV light. One can also see the development of PI clusters elongated in the direction perpendicular to the rubbing direction, showing that morphological anisotropy has changed.

IV. CONCLUDING REMARKS

We have described the physical origin of surface modification of the PI layer caused by exposure to UV light. The photomodified unrubbed PI surface shows weak anisotropy between the directions parallel and perpendicular to LPUV light's polarization direction, which suggests that the morphological anisotropy plays a very important role in LC alignment. In the case of rubbed PI films subsequently exposed to RPUV light, it was found that macroscopic scratch lines are inconsequential, and that the morphological anisotropy at a submicrometer length scale is important for LC alignment. The fine tuning of LC alignment can be achieved by combining rubbing and controlled LPUV exposure of PI films. The surface morphological anisotropy produced by rubbing is reduced by UV exposure through the photodissociation process. Details of the competition between the effects of rubbing and LPUV exposure deserve further investigations. A simple model presented here can be used to describe physical phenomena associated with photochemical processes in polymer system.

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