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Abstract- This report presents the surface acoustic wave (SAW) mediated realignment for molecular orientation of polyimide films in curing process. The order parameters of polyimide films were obtained by measuring the anisotropic infrared absorbance using polarized infrared absorption spectroscopy. Experimental results of the double layered specimens indicated that the monomer chains were aligned to parallel to the wave fronts of SAW. The double layered specimens were made of a well cured polyimide film subsequently coated with the second film. The first film provides attractive forces to anchor the molecules of the second polyimide film on the first film. In the present experiments, the molecular orientation order parameter of SAW processed polyimide film is 0.23. It is between the values of unrubbed film (0.069) and rubbed one (0.535).

Keywords- surface acoustic wave, molecular orientation, polyimide, alignment layer, FTIR

I. INTRODUCTION

The application of surface acoustic waves (SAW) and other guided acoustic modes in realignment of nematic liquid crystal (NLC) molecules has received attention for many years [1-5]. However, the need for relatively large actuating area limits its industrial applications. Instead of switching on/off the nematic liquid crystals, surface acoustic wave mediated alignment could be used for fabrication of large area alignment films in liquid crystal based flat panel displays. Polyimide is the most common material used in such alignment films. In addition, the flexible display is built on a variety of plastics. Most fabrication processes commonly used for flat panel displays can not be directly transferred to flexible devices.

Mechanical rubbing is a powerful technique used in polyimide films spin coated on a substrate for aligning liquid crystal molecules along the rubbing direction. The alignment mechanism of the liquid crystal molecules on the rubbed polyimide films is not clearly understood. It is believed that the molecular orientation of the rubbed polyimide films plays an important role in the liquid crystal molecular alignment [6-8].

Surface acoustic waves have been employed to manipulate micrometer sized particles suspended in a fluid and in many applications of microfluidics [9-10]. Ultrasonic manipulation comprises acoustic radiation and acoustic streaming. The former is resulted from acoustic pressure wave propagating in the fluid and the latter is due to the second-order effect caused by leaky acoustic waves and free surface effect. The acoustic radiation leaked into the fluid induces a thickness depending recirculation in the fluid.

The thermal imidization of polyamic acid precursors into polyimide needs heating the precursors to temperature ranging from 250 to 400°C [12]. The surface acoustic wave mediated alignment could assist ring closure proceeded by dehydration in polyamic acid to yield polyimide. The surface acoustic wave process can provide further energy to assist curing of polyimide films and requires no such high temperature.

At present mechanical rubbing can achieve very good quality unidirectional molecular orientation in the rubbed polyimide films. However, it also generates a large number of waste powders and requires additional cleaning. Researchers are still looking for other non-contact techniques to align molecular orientation in the polyimide films, such as photopolymers, nitrogen plasma beam, ion-beam, etc. This paper presents the study on surface acoustic wave mediated alignment for the curing process of polyimide films.

II. EXPERIMENT

Fig. 1 shows the experimental setup of surface acoustic wave processed alignment for curing of polyimide film. The specimen spin-coated with polyamic acid/solvent is placed on a temperature controllable planar heater. A glass waveguide having a wedge tip of 15° apex angle at one end contacts the top surface of the specimen with 30° inclined angle. The displacement amplitude of
surface acoustic waves generated from the SAW device attached on the glass waveguide can be amplified if the acoustic waves transmit through the wedge tip. A lift jack is used to sustain the incline of the glass waveguide.

In experiment, an interdigitated transducer (IDT) made on a 128-deg-rotated Y-cut lithium niobate wafer was used to launch surface acoustic waves propagating along the X direction. The surface acoustic waves were launched by a 16 MHz IDT. A standing wave orientated along the X direction was formed by the combination of forward and reflected surface acoustic waves. The pressure gradient induced among the wave crests and troughs can cause long chained molecules, such as polyamic acid, polyimide, etc., aligned in the direction parallel to the nodal lines if a large number of acoustic waves are used to drive the alignment.

The specimens were made of the PIA-5370-33C solvent coating on surface polished calcium fluoride substrates. The spin-coated polyimide specimen was 25.4×25 mm. The PIA-5370-33C solvent comprises 5 wt % polyamic acid, 30 wt % N-methy-2-pyrrolidone, 35 wt % Butyl cellosolve, and 30 wt % γ-Butyrolactone. Two kinds of specimens were made. The first sets of specimens were spin coated with a single layer. The second ones were coated with two layers. The first layer had been heated up to 200°C such that the polyamic acid was transferred into polyimide. The second layer was then coated with solvent for later curing. The first cured polyimide films were used to provide anchoring forces to the molecules in the second polyimide films.

The molecular orientation of polyimide films is of difficult to be measured by common equipment because of its very thin thickness. Polarized IR absorption spectroscopy is an effective method for determining the anisotropic property of polyimide films via polarization angle dependence. The IR light polarized parallel to the polarization direction of an IR active vibration is absorbed. According to the polarization angle dependence at normal incidence, the distribution of molecular orientation in a film can be determined.

III. RESULTS

Fig. 2 shows the absorption spectra of the unrubbed and SAW processed polyimide films at curing temperatures of 200°C and 250°C. Each absorbance curve has four significant bands, including the C-O-C asymmetric stretching vibration at 1,246 cm⁻¹, the C-N stretching vibration of the (OC)₂NC bond at 1,375 cm⁻¹, the phenyl C-C stretching vibration at 1,500 cm⁻¹, and the C = O asymmetric stretching vibration at 1,724 cm⁻¹, respectively. The first three vibration modes are polarized parallel to the monomer chain. The higher curing temperature certainly induces better absorbance. The evidence indicates that SAW processed polyimide films significantly achieve higher absorbance than the unrubbed ones at either 200°C or 250°C curing temperature.

The polarized IR absorbance of single layered specimens processed by SAW was below noise level. There was no evidence that molecular orientation of polyimide films was aligned by SAW. However, the double layered specimens had very clear anisotropic IR absorbance. The first layer of well-cured polyimide provided enough anchoring force to one end of each molecule such that the acoustic pressure gradient among wave crests and troughs can align the remaining part of the molecule before the liquid solvent evaporated. In the latter experiment, we only used the double layered specimens for comparing anisotropic infrared absorbance.

Fig. 3 shows the polarization angle dependence of the 1,500 cm⁻¹ band at normal incidence. The 1,500 cm⁻¹ band is assigned to the phenyl C-C stretching vibration. As mentioned before, it is polarized parallel to the monomer chain. The solid circles indicate the data points for un rubbed, rubbed and SAW processed polyimide films. For the rubbed film, the anisotropy shown in Fig. 3(b) indicates that the monomer chains of polyimide are parallel to the rubbing direction. However, the monomer chains of polyimide film are perpendicular to the direction of acoustic wave propagation. In other words, the monomer chains become parallel to the nodal lines of the standing wave. The molecular reorientation is caused by pressure gradient among the wave crests and troughs.
The acoustic streaming has no significant influence on the thin polyimide films during curing process although the bulk fluid recirculation could cause polyimide molecules non-uniform aligning in the regions near the edges of specimen. This acoustic streaming effect could be further reduced if thin spin-coating liquid polyamic acid coating, surface acoustic waves of shorter wavelength, or larger polyimide films are considered.

IV. DISCUSSION

The order parameter [6] is used to evaluate the anisotropic property of polyimide layer. It is of the form

\[ S = \frac{A_{||} - A_{\perp}}{A_{||} + 2A_{\perp}} \]  

in which \( A_{||} \) and \( A_{\perp} \) are defined as the IR absorbance measured when the polarization of an incident light parallel or perpendicular to the monomer chain, respectively. The order parameters are 0.069, 0.535, and 0.230 for unrubbed, rubbed, and surface acoustic wave processed polyimide films. The average value of polarization angle dependence is proportional to the thickness of polyimide film. For unrubbed film the average value of absorbance is larger than those of rubbed and SAW processed films. In addition, the order parameter is a significant measure can be used to distinguish the degree of anisotropy for polyimide film.

The polyimide film can be formed in lower...
temperature than usual by using the SAW mediated alignment process on the polyimide films. The alignment is feasible for large area flexible substrates. Further studies are needed although the present method is possible to provide good liquid crystal properties and mechanical stability for the flexible liquid crystal displays.

ACKNOWLEDGMENT

The authors would like to thank Prof. H.-M. Chen of Department of Photonics at NCTU for helpful discussions.

REFERENCES


一、會議概要


會議論文分為五大領域：

1. 醫用超音波學(Medical Ultrasonics)
2. 感測器、非破壞評估及工業應用(Sensors, NDE & Industrial Applications)
3. 物理超音波學(Physical Acoustics)
4. 微聲學(Microacoustics – SAW, FBW, MEMS)
5. 換能器及其材料(Transducers and Transducer Materials)

各領域下又區分為 4 至 11 個主題進行討論。該會議中心場地寬闊，大會同時使用 6 間會議室進行口頭報告與討論，每間會議室可容納約 150-250 人，每篇論文的發表及討論時間共 15 分鐘，遇缺席者則空下該時段，方便與會者進出各個研討會場，選擇聆聽有興趣的論文。會議論文總共有口頭發表者 371 篇、海報發表者 504 篇，其中來自台灣的論文共有 26 篇。另外在會場闢有廠商展覽及論文海報張貼發表場地，相較於論文海報區，儀器商的展覽場卻較為冷清。會議設有 Gala Award Reception 及正式晚宴，於 9 月 21 日晚上 8:00-11:00 在 Ergife Palace Restaurant 的 Le Quattro Stagioni 廳舉行，除了主辦單位的致詞，還有會士授證及頒獎儀式。


本篇論文為國科會計畫 NSC 97-2212-E-009-009 的研究成果，應用表面聲波操控液晶面板的配向膜聚亞醯胺(polyimide)分子的排列，根據傅立葉轉換紅外線光譜(Fourier transform infrared spectroscopy，簡稱 FTIR)的檢測數據顯示，表面聲波輔助聚亞醯胺分子排列試片的秩序參數為 0.23，不遜於昂貴的電漿配向。目前業界採用之毛刷(rubbing)配向的秩序參數可達 0.535，但是容易沾附纖毛、產生碎屑，必須進行二次處理，以去離子水

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洗淨、烘乾，增加成本。聚亞醯胺的強度高，無法直接塗佈於面板表面，一般是將其前驅物聚醯胺酸(polyamic acid)溶液塗佈於試片表面，再加熱烘烤至200-250°C，使其去水、環化，轉化成聚亞醯胺。本研究採用氟化鈣作為配向膜的基材，於聚醯胺酸的加熱過程中，將表面聲波以玻璃波導傳送至試片，與邊緣反射波干涉形成駐波，聲波的壓力分佈(acoustic radiateon)會造成鍊狀高分子平行於波前排列。表面聲波也會產生聲流(acoustic streaming)，造成聚醯胺酸溶液流動，因此，本文大膽採用雙層配向膜設計，先塗佈、烘烤第一層聚亞醯胺薄膜，以氫鍵吸附第二層的聚醯胺酸分子，類似插秧的方式固著分子，再以表面聲波使其排列方向整齊平行於波前，大幅改進配向的效果。該篇報告獲得與會者的高度興趣與迴響。

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1. Film Bulk Acoustic Resonator (FBAR)
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3. Microwave Acoustic Sensors
4. Therapeutic Ultrasound
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6. Estimation and Imaging of Blood Flow Velocity
7. SAW Modeling Techniques
8. Piezoelectric Ultrasound Transducer Fundamentals – Materials, Structure, Behavior, and Analysis
9. Quantitative Acoustic Microscopy – Fundamentals and New Applications from Cells to Airplanes
10. Ultrasound Imaging Systems: from Principles to Implementation
11. Passive UHF RFID Tags, Systems, and Applications
12. Ultrasonic Signal Processing for Detection, Estimation, and Imaging
13. Time Reversal Acoustics
14. Ultrasound Contrast Agents: Theory and Experiment
15. Zooming into the Near Field
16. CMUTs: Theory, Technology, and Applications
17. Guided SH-SAW Devices for Liquid-Phase Biochemical Sensors
18. Elasticity Imaging: Dynamic Approaches

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Effect of Surface Acoustic Waves on Curing of Polyimide Films

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Figure 1. Schematic view of surface acoustic wave processed alignment for curing of polyimide film. 1- specimen coated with polyimide film, 2- glass waveguide with wedge tip, 3- interdigitated transducer, 4- planar heater, 5- lift jack.
having a wedge tip of 15° apex angle at one end contacts the top surface of the specimen with 30° inclined angle. The displacement amplitude of surface acoustic waves generated from the SAW device attached on the glass waveguide can be amplified if the acoustic waves transmit through the wedge tip. A lift jack is used to sustain the incline of the glass waveguide.

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The molecular orientation of polyimide films is of difficult to be measured by common equipment because of its very thin thickness. Polarized IR absorption spectroscopy is an effective method for determining the anisotropic property of polyimide films via polarization angle dependence. The IR light polarized parallel to the polarization direction of an IR active vibration is absorbed. According to the polarization angle dependence at normal incidence, the distribution of molecular orientation in a film can be determined.

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Fig. 2 shows the absorption spectra of the unrubbed and SAW processed polyimide films at curing temperatures of 200°C and 250°C. Each absorbance curve has four significant bands, including the C-O-C asymmetric stretching vibration at 1,246 cm⁻¹, the C-N stretching vibration of the (OC)₂NC bond at 1,375 cm⁻¹, the phenyl C-C stretching vibration at 1,500 cm⁻¹, and the C = O asymmetric stretching vibration at 1,724 cm⁻¹, respectively. The first three vibration modes are active vibration is absorbed. According to the polarization angle dependence at normal incidence, the distribution of molecular orientation in a film can be determined.

![Figure 2. FTIR spectra of unrubbed and SAW processed polyimide films under curing temperatures up to 200°C and 250°C.](image)

The polarized IR absorbance of single layered specimens processed by SAW was below noise level. There was no evidence that molecular orientation of polyimide films was aligned by SAW. However, the double layered specimens had very clear anisotropic IR absorbance. The first layer of well-cured polyimide provided enough anchoring force to one end of each molecule such that the acoustic pressure gradient among wave crests and troughs can align the remaining part of the molecule before the liquid solvent evaporated. In the latter experiment, we only used the double layered specimens for comparing anisotropic infrared absorbance.

Fig. 3 shows the polarization angle dependence of the 1,500 cm⁻¹ band at normal incidence. The 1,500 cm⁻¹ band is assigned to the phenyl C-C stretching vibration. As mentioned before, it is polarized parallel to the monomer chain. The solid circles indicate the data points for unrubbed, rubbed and surface acoustic wave processed polyimide films. For the rubbed film, the anisotropy shown in Fig. 3(b) indicates that the monomer chains of polyimide film are perpendicular to the rubbing direction. However, the anisotropy shown in Fig. 3(b) indicates that the monomer chains of polyimide film are parallel to the direction of acoustic wave propagation. In other words, the monomer chains become parallel to the nodal lines of the standing wave. The molecular reorientation is caused by pressure gradient among the wave crests and troughs.

### IV. DISCUSSION

The order parameter [6] is used to evaluate the anisotropic property of polyimide layer. It is in the form

$$ S = \frac{A_\perp - A_\parallel}{A_\parallel + 2A_\perp} \quad (1) $$

in which $A_\perp$ and $A_\parallel$ are defined as the IR absorbance measured when the polarization of an incident light parallel or
The acoustic streaming effect could be reduced if thin spin-uniformly aligning in the regions near the edges of specimen. Bulk fluid recirculation could cause polyimide molecules non-uniformly aligning in the bulk fluid recirculation during curing process although the thin polyimide films during curing process. The acoustic streaming has no significant influence on the average value of absorbance is larger than those of rubbed and SAW processed films. In addition, the order parameter is a significant measure can be used to distinguish the degree of anisotropy for polyimide film.

The acoustic streaming has no significant influence on the thin polyimide films during curing process although the bulk fluid recirculation could cause polyimide molecules non-uniformly aligning in the regions near the edges of specimen. This acoustic streaming effect could be further reduced if thin spin-coating liquid polyamic acid coating, surface acoustic waves of shorter wavelength, or larger polyimide films are considered.

The polyimide film can be formed in lower temperature than usual by using the SAW mediated alignment process on the polyimide films. The alignment is feasible for large area flexible substrates. Further studies are needed although the present method is possible to provide good liquid crystal properties and mechanical stability for the flexible liquid crystal displays.

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Figure 3. Polarization angle dependences of the 1500 cm⁻¹ band for (a) unrubbed, (b) rubbed and (c) surface acoustic wave processed polyimide films. The arrowheads indicate the directions of rubbing in (b), and surface acoustic waves propagating in (c).

