Patterned photoresist spacers and photo-induced alignment coatings for liquid crystal waveplates and polarizers

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ABSTRACT

The OMEGA laser system uses waveplates and polarizers to alter the polarization of the laser light. Both devices are formed using two glass substrates that contain alignment layers and are separated by vacuum-deposited thin-film spacers and filled with liquid crystals (LCs). Vacuum deposition of thin-film spacers is time-consuming and requires the use of expensive equipment. In this study, spacers were formed by photopatterning of a negative photoresist material using standard photolithographic techniques. These photoresist spacers are comparable to thin-film spacers in quality and are much easier to produce. A new non-contacting method for generating uniform LC alignment by irradiation of a linearly photopolymerizable polymer (ROLIC ROP-203/2CP) with polarized UV light was also investigated as an alternative to the current mechanical buffing process, which leaves particles and static charges on the substrate. This "photobuffing" process is easily scalable to large apertures. Because the application of photoresist spacers does not impact the laser damage threshold of the optics and the ROLIC photoalignment material has high laser induced damage resistance, both materials are feasible for use in the laser system.

1. INTRODUCTION

1.1 Vacuum deposition of thin-film spacers

The current process for manufacturing liquid crystal waveplates (LCWs) involves vacuum depositing thin-film spacers onto the inner surface of each glass substrate.¹ When the LC cell is assembled, the spacers create a gap between the two substrates, which is then filled with LC material. These spacers are formed by depositing a spacer coating in even layers on the surface of the substrates until the desired spacer thickness is attained. A spacer coating run may take four to six hours to produce the specified thickness of $6.5 \pm 0.23 \,\mu$ m for an LCW.² Several coating runs may be necessary to ensure the spacer thickness is correct. This process takes a fair amount of coater time in set-up and operation. Thus, vacuum deposition of thin-film spacers is time-consuming and requires the use of expensive equipment, including large vacuum chambers.

1.2 Mechanical buffing of alignment layers

Liquid crystal waveplates are filled with LCs, which alter the polarization of light when all the LC molecules are properly aligned along the same axis of polarization, facing the same direction. To align the LC molecules, the inner surface of each glass substrate is spin-deposited with a nylon coating and then mechanically buffed in a single direction to create microscopic grooves in the surface of the coating.² After the cell is constructed and filled, the LC molecules will orient themselves along the grooves, resulting in uniform alignment. However, this "rubbing" technique that is used to align the LCs leaves unwanted particles and static charges on the substrate.³ The

mechanical buffing procedure also utilizes costly equipment that requires set-up and must be maintained (Fig. 1).

2. EXPERIMENT

2.1 Substrate preparation

In order for the spacer and alignment coatings to adhere to the substrates, the surfaces of each substrate must be properly cleaned and prepared. In this study, substrates were first wetted and scrubbed using 0.22 μ m filtered water and 0.05 μ m deagglomerated alumina polish until the water broke on the surfaces of the substrates. The substrates were then thoroughly rinsed and cleansed in an ultrasonic bath for 60 minutes (Fig. 2). After cleansing, substrates were rinsed with deionized water, dried using a nitrogen air gun, and baked at 90°C on a covered hotplate for 60 minutes.

2.2 Patterned photoresist spacers

In this study, spacers were formed by photopatterning of a negative photoresist material using standard photolithographic techniques⁴ (Fig. 3). To obtain maximum process reliability, the substrates were baked at 200°C on a covered hotplate for 5 minutes, thereby ensuring that the surfaces were clean and dehydrated. Next, the surfaces of the substrates were covered with Microchem SU-8 2010. SU-8 is a negative photoresist, which, after exposure to a UV light source, becomes insoluble when developed. The coated substrates were then spun under cover at 2500 rpm for 60 seconds to produce a 12 μ m step height for the spacers. After spin-coating, substrates were pre-baked at 65°C on a covered hotplate for 1 minute, then softbaked at 95°C for 2 minutes. Before UV illumination, a metal photoresist spacer mask was positioned and secured over the coated side of

each substrate. The mask contained small holes that would allow some areas of the coating to be exposed to the light source, resulting in four uniformly sized spacers on the surface of the substrate. The substrates were then illuminated under a UVP mercury vapor lamp, coated sides facing upward, for 18 seconds (Fig. 4). Following exposure, substrates were post-exposure baked on a covered hotplate at 65°C for 1 minute, and at 95°C for 2 minutes. Then, substrates were allowed to stand for 10 minutes before developing.

The SU-8 photoresist was developed using an immersion develop process. Substrates were completely submerged with the coated sides facing upward, in SU-8 Developer fluid for 3 minutes (Fig. 5). Strong agitation was applied while developing to ensure maximum resist removal. Afterwards, substrates were rinsed using isopropanol for 5-10 seconds, and then rinsed briefly using deionized water. Substrates were then dried using a nitrogen air gun and hard baked at 150°C on a covered hotplate for 10 minutes. Overall, the process for photopatterning resist spacers takes about 30-40 minutes.

2.3 Photo-induced alignment coatings

As an alternative to mechanical buffing, the process of "photobuffing" was investigated as a cleaner, non-contacting method for generating uniform LC alignment across the surface of a substrate. First, substrates were rinsed using deionized water and dried using a nitrogen air gun. Substrates were not wiped dry to prevent damage of the photoresist spacers. Next, substrates were heated at 110°C on a covered hotplate for 60 minutes to remove any remaining moisture before being spin-coated. Once dry, substrates were coated with filtered cyclohexanone using a 0.45 µm PTFE 4 mm hydrophobic-

filtered syringe. The spin-coater was covered using a glass dish for 2 minutes before spin-coating, in order to fill the ambient with cyclohexanone, providing for a more even alignment coating. The cyclohexanone was then spun off for 15-20 seconds (Fig. 6).

About 1/3 mL (or enough to cover the surface of the substrate) of 1-25% diluted ROLIC ROP-203/2CP was uniformly distributed onto the surface of each substrate using a 0.25 µm filtered syringe. ROLIC is a linearly photopolymerizeable polymer (LPP), which, when irradiated using a polarized UV light, will polymerize along the direction of polarization of the light source. Once a device is assembled and filled with LCs, the LC molecules will orient themselves along the polymer chains in the LPP, producing alignment without leaving any residue, unlike the mechanical buffing process.⁵

After dispensing the ROLIC material onto the substrates, the spin-coater was covered to decrease evaporation of the coating material while substrates were allowed to stand for 30 seconds. Next, substrates were spun under cover at 3000 rpm for 60 seconds, and then placed on a covered hotplate at 130°C for 10 minutes. Substrates were removed from the hotplate and left to cool for 60 minutes in a clean area. Finally, the substrates were set in a metal holding chamber and exposed to polarized 365-nm light from a xenon source at 65 watts for 10 minutes each. The substrates were positioned behind a Schott UG-11 light filter and a "pile-of-plates" silica polarizer set at Brewster's angle, resulting in linear polarization of the ROLIC-coated surfaces (Fig. 7).

2.4 Cell assembly

Following the application of photoresist spacers and photoalignment material, substrates were assembled together to create a cell. Only one substrate in each cell contained spacers (structured substrate). To assemble a cell, a very small amount of UV epoxy was applied to each spacer of a structured substrate. The coated surface of the structured substrate was then carefully aligned with the coated surface of a non-structured substrate, and gentle pressure was applied to ensure good contact. The substrates were aligned with antiparallel polarizations, so that the cell should act as a waveplate when viewed under crossed polarizers. The cell was then exposed under a UVP mercury vapor lamp for 1 minute with a weight placed on top to maintain constant pressure on the epoxy. Once the epoxy was cured, a vertical fill technique was used to fill the cell with the nematic liquid crystal E7 by Merck. The empty cell was held vertically and slowly filled by dispensing drops of LC across the top edge of the cell using a filtered syringe (Fig. 8). A vertical fill technique was used because it is generally more successful than a horizontal fill in creating uniform fills. The filled cell was then observed under crossed polarizers to assess the quality of alignment. The device was sealed around the edge using 5-Minute Epoxy. After allowing the epoxy to fully harden for 15-20 minutes, the finished device was cleaned using acetone.

3. RESULTS

3.1 Liquid crystal cell results

The goal of this research was to study the feasibility of using patterned photoresist and photoinduced alignment coatings to replace thin-film spacers and buffed nylon coatings, respectively, for use in LCWs. The LC cells that were made had non-uniform polarization, which resulted in light and dark patches of LCs when viewed under crossed polarizers. The xenon light source that was used to polarize the alignment coatings was observed to have imperfect polarization. This resulted in the poor polarization of the ROLIC coatings. The "pile-of-plates" polarizer in the set-up was adjusted to 56.1° to create better alignment.

Despite readjustment of the UV light set-up, the LC cells that were made still lacked uniform alignment. The ROLIC coating on the substrates proved to be too thick and unevenly distributed, producing microscopic ridges that diffracted the light hitting the surface, thereby disrupting polarization. In addition, the inner surface of the irradiation set-up was not completely black, which caused the UV light to scatter and become depolarized. To resolve this, the inside of the set-up and metal holding chamber were resealed using black tape. Overall, high quality alignment of the ROLIC material was achieved over a 1" by 1" area of the coated surface.

3.2 Photoresist and ROLIC material damage test data

Damage tests demonstrated that both the SU-8 photoresist and the ROLIC photoalignment materials are viable for use in the OMEGA/EP laser systems. Substrates with photoresist spacers were

cleansed repeatedly in an ultrasonic bath to determine the durability of the spacers. After multiple washes, the photoresist spacers proved to be highly wash-resistant, and no visible damage was recorded. The spacer step height is reproducible, and can be easily altered by adjusting the speed and/or time of spin-coating the resist. The ideal step height for this experiment was 12.0 μ m, and the spacers produced averaged about 11.7 μ m when spun at 2500 rpm for 60 seconds (Fig. 9). Damage testing of substrates with SU-8 resist spacers showed no residue on the clear aperture in the center of the substrates. This demonstrates that cells made using photoresist spacers will be high quality, and will not encounter any interference from excess resist material left in the clear aperture.

High quality samples of the ROLIC photoalignment material were prepared on DTO substrates in order to test the near IR laser damage resistance of the alignment coating. Damage tests were conducted on both samples that had been exposed to low intensity, continuous wave (CW) polarized UV light and samples that were not exposed to any UV to determine whether there was any reduction in the laser damage threshold as a result of the CW irradiation with polarized UV. Damage tests were conducted using a 0.5 mm spot size and a 1 ns pulse at 1054 nm. The 1-on-1 near IR laser damage thresholds of ROLIC samples that had been exposed to low energy, CW polarized UV light for 10 minutes was 27.4 \pm 4.6 J/cm² for defect-containing test sites, and 57.3 \pm 1.5 J/cm² for defect-free sites. The near IR laser damage threshold for an N-on-1 test was 30.0 \pm 7.7 J/cm² for sites with defects. For an unexposed ROLIC sample, the 1-on-1 and N-on-1 damage thresholds were 45.8 \pm 5.0 J/cm² and > 43.0 J/cm², respectively. Surprisingly, the laser induced damage resistance of the ROLIC material exceeded that of nylon (14.15-15.81 J/cm²), and was equivalent to that of bare fused silica (30-50 J/cm²).⁶

4. CONCLUSIONS

4.1 Application of patterned photoresist spacers

Photoresist spacers have proven to be much easier than thin-film spacers to develop and apply. The time it takes to make spacers can be reduced from 4-6 hours to 30-40 minutes by photopatterning of an SU-8 resist in a single coating run, as opposed to multiple coating deposits. An additional benefit of the photoresist process is the lack of expensive coating equipment to set-up and maintain. Photoresist spacers are comparable to thin-film spacers in quality, but more research needs to be done to determine the thickness error between spacers on a single substrate.¹ The uniformity of the spacers on each substrate is critical to the quality of the finished device (Fig. 10).

4.2 Application of ROLIC photoalignment material

The high near IR laser damage thresholds of the ROLIC ROP-203/2CP material show that it is feasible for use in the laser system. The alignment coating can be applied using a spin-coating method, with which the coating thickness can be easily adjusted as needed. Following this study, it is worth developing and testing other photoalignment materials in future studies.⁶

4.3 Continued development of the photobuffing process

Photobuffing has the potential to become more convenient and efficient than the current process of mechanical buffing. Non-contact photobuffing of the photoalignment material eliminates residue and is easily scalable to large areas. This study was unable to produce high quality alignment over a large area due either to irradiation non-uniformities or mechanical damage to the alignment coatings

during device assembly. Once uniform alignment is achieved over a greater area, the photobuffing process can be used in combination with a photoresist mask to create cells with patterned directional alignment in multiple, separate alignment domains. Further studies can be done to reduce regions of reverse twist and boundary disclinations, and to reduce surface and coating flaws.³

4.4 Future studies

The next logical step in developing the processes of photobuffing and patterned photoresist spacers would be to make an actual filled LC device for damage testing. The device could be measured for performance as a half-waveplate, taking into account the uniformity and quality of the results.¹ Photobuffing opens scientific avenues to photopatterning through the use of a mask, and its potential application in making modulators, switchable devices, and diffraction gratings.

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REFERENCES

- [1] Rigatti, Amy. Private communication (2007).
- [2] *Manufacturing Procedure, Liquid Crystal Optics*, F-FJ-P-01 Rev. A OMEGA Upgrade, p. 17-33, Laboratory for Laser Energetics, Rochester: University of Rochester, 18 Jan. 1993.
- [3] A. R. White. *Photopatterning of Liquid Crystal Alignment Cells*. Laboratory for Laser Energetics, Rochester: University of Rochester, 25 Aug. 2004.
- [4] Microchem. NANOTMSU-8 Negative Tone Photoresist Formulations 2-25. Newton: Feb. 2002.
- [5] Myhre, Graham. "Updated SU-8 2010/Staralign Process." Laboratory for Laser Energetics, Rochester: University of Rochester, 25 May 2005.
- [6] Marshall, Kenneth L. Private communication (2007).

FIGURES



Figure 1. Set-up of a mechanical buffer.



Figure 2. Cleaning of substrates in an ultrasonic bath.



Figure 3. Diagram of spacer arrangement on a fused silica substrate.



Figure 4. Illumination of substrate under a mercury vapor lamp, showing mask with designated areas where spacers will be exposed.



Figure 5. Immersion development process using SU-8 Developer to remove unexposed resist.



Figure 6. Spin-deposition of ROLIC photoalignment coating. The spin coater is covered with a glass dome to prevent rapid evaporation of the solvent during the deposition process.



Figure 7. Schematic diagram of contact-free photobuffing process.



Figure 8. A vertical fill technique used to fill a cell with liquid crystals.

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Figure 9. Zygo NewView microinterferometer data for a patterned photoresist spacer (step height: 11.7 μm).

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Figure 10. Zygo NewView microinterferometer data for a patterned photoresist spacer (step height: 15.5 μm).