This article was downloaded by: [University of California, San Diego]

On: 09 August 2012, At: 14:21 Publisher: Taylor & Francis

Informa Ltd Registered in England and Wales Registered Number: 1072954 Registered office: Mortimer House, 37-41 Mortimer Street, London W1T 3JH,

UK



# Molecular Crystals and Liquid Crystals

Publication details, including instructions for authors and subscription information: <a href="http://www.tandfonline.com/loi/gmcl20">http://www.tandfonline.com/loi/gmcl20</a>

### Integration of Optical Pumped Dye Laser on Organic Microflowcytometry Chip

Yuji Oki <sup>a</sup> , Yukinori Ogawa <sup>a</sup> , Kenichi Yamashita <sup>b</sup> , Masaya Miyazaki <sup>b</sup> & Mitsuo Maeda <sup>c</sup>

<sup>a</sup> Graduate School of Information Science and Electrical Engineering, Kyushu University, Fukuoka, Japan

<sup>b</sup> National Institue of Advanced Industrial Science and Technology Kyushu, Shuku, Tosu, Saga, Japan

<sup>c</sup> Kurume National College of Technology, Komorino, Kurume, Japan

Version of record first published: 12 Mar 2007

To cite this article: Yuji Oki, Yukinori Ogawa, Kenichi Yamashita, Masaya Miyazaki & Mitsuo Maeda (2007): Integration of Optical Pumped Dye Laser on Organic Microflowcytometry Chip, Molecular Crystals and Liquid Crystals, 463:1, 131/[413]-140/[422]

To link to this article: http://dx.doi.org/10.1080/15421400601027684

#### PLEASE SCROLL DOWN FOR ARTICLE

Full terms and conditions of use: <a href="http://www.tandfonline.com/page/terms-and-conditions">http://www.tandfonline.com/page/terms-and-conditions</a>

This article may be used for research, teaching, and private study purposes. Any substantial or systematic reproduction, redistribution, reselling, loan,

sub-licensing, systematic supply, or distribution in any form to anyone is expressly forbidden.

The publisher does not give any warranty express or implied or make any representation that the contents will be complete or accurate or up to date. The accuracy of any instructions, formulae, and drug doses should be independently verified with primary sources. The publisher shall not be liable for any loss, actions, claims, proceedings, demand, or costs or damages whatsoever or howsoever caused arising directly or indirectly in connection with or arising out of the use of this material.

Mol. Cryst. Liq. Cryst., Vol. 463, pp. 131/[413]-140/[422], 2007

Copyright © Taylor & Francis Group, LLC ISSN: 1542-1406 print/1563-5287 online DOI: 10.1080/15421400601027684



## Integration of Optical Pumped Dye Laser on Organic Microflowcytometry Chip

#### Yuji Oki Yukinori Ogawa

Graduate School of Information Science and Electrical Engineering, Kyushu University, Fukuoka, Japan

#### Kenichi Yamashita Masaya Miyazaki

National Institue of Advanced Industrial Science and Technology Kyushu, Shuku, Tosu, Saga, Japan

#### Mitsuo Maeda

Kurume National College of Technology, Komorino, Kurume, Japan

Integration techniques of tunable film dye laser on a plastic optical application chip were studied. The developed film dye lasers were waveguided distributed feedback (DFB) lasers, and they can cover the wavelength from 400 to 1100 nm. We fabricated microflowcytometry chip integrated with DFB film lasers as a first example. Partly film-coating technique and film lithography fabrication with an excimer laser were tried and combined each other. The film DFB dye laser was found as an useful technique for multi-color laser integration, and they showed comparable performance our traditional spin-coated film dye lasers.

**Keywords:** integration; organic microflowcytometry chip; plastic optical application chip; tunable film dye laser

#### 1. INTRODUCTION

Plastic dye laser have been studied by many research groups [1–3], and waveguided and/or a distributed feedback (DFB) solid-state dye lasers have also been studied [4–8] to simplify tunable laser systems and to decrease laser threshold energy.

Address correspondence to Yuji Oki, Graduate School of Information Science and Electrical Engineering, Kyushu University, Fukuoka 812.8581, Japan. E-mail: oki@ed. kyushu-u.ac.jp

132/[414] Y. Oki et al.

We study optically pumped waveguide-DFB film dye lasers as integratable tunable lasers. Laser-integration techniques are very important to extend applicable region of tunable lasers. We developed distributed-feedback waveguide lasers based on acrylic co-polymer doped with laser dyes, and demonstrated conversion efficiency as high as 10% [9], laser threshold less than  $\mu J$ , wide-tunability [10], extended durability [11], tunable random medium lasers [9], and also novel pumping scheme for fiber top laser alignment [12].

Recently, we suggest integrations of the waveguide DFB laser films on chip devices, such as biological analyzing chips, bio-sensors, medical diagnostic chips, and microflowcytometry chips. As a first trial, we are developing laser integrated plastic flow-chips. They are mainly made of polymers and integrates multicolor laser waveguides that can be selected from covered wavelength region over 400 nm to 1100 nm.

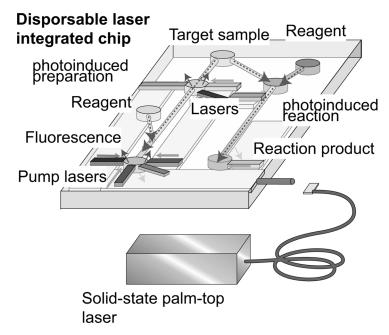
### 2. CONCEPT OF LASER INTEGRATED FLOWCYTOMETRY CHIP

Figure 1 shows conceptual scheme of the integrated microflowcytometry plastic chip. The reservoirs for sample, reagent and reaction product are connected with flow-channel. An electrophoresis can be used for example. Multicolor film lasers are located on the side of the flow channel to irradiate the flowing target directly. The wavelength of the lasers are determined by the requirement of measurements of the each part. For instance, the sample from reservoir was monitored and prepared with the laser irradiation first before mixing with reagents. At the mixing point, photo-induced reaction is also possible. In result, the reaction product was optically investigated by laser irradiation. Locating many lasers along to the flowchannel can measure temporal change of the reaction product. The lasers are pumped by a fiber-guided pumping pulses through an optical pumping delivery network mode of optical waveguides. Each integrated laser can oscillate at individual wavelength, so multiple reagents can be used simultaneously with a pumping laser pulse of single wavelength.

#### 3. SILICA CAPILLARY MOUNTED DFB LASER CHIP

#### 3.1. Fabrication and Experiment

As a trial of the development and preliminary experiment, a silica capillary and a V-groove on the film laser chip was fabricated as shown



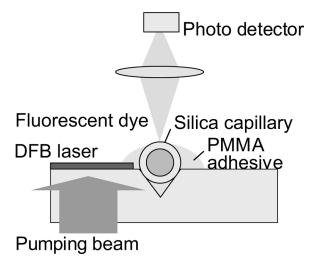
**FIGURE 1** Conceptual schematic of an integrated tunable laser system on a chip.

in Figure 2. 20 of Rhodamine640 doped DFB laser waveguide in array formation was fabricated with the procedure similar to previous work [13]. After carving a V-groove on the surface of the chip, a silica capillary (i.d. and o.d. were 500 and 660  $\mu m$ , respectively) was mounted by using a PMMA adhesive. PMMA adhesive also filled the optical path between the waveguide's end and the side wall of the capillary to avoid the scattering. The pumping laser was a passively Q-switched Cr:Nd:YAG laser SHG (Uniphase, PNG-002025-040, 532 nm, 30  $\mu J$ , 0.5 ns FWHM), and the pumping beam was sheet-formed and injected from a bottom surface of the chip. The output of the pumped DFB laser comes from both ends, and one of them was coupled directly to the capillary and it pumped the internal solution. The fluorescence from the capillary was collected by the lens and detected by the monochromater (Solar TII, MS7504, with a CCD camera LD 2048).

#### 3.2. Results and Discussion

Figure 3 shows an example of the fluorescence signal from Nileblue690 dye's solution in the capillary. The Nileblue690 has a similar

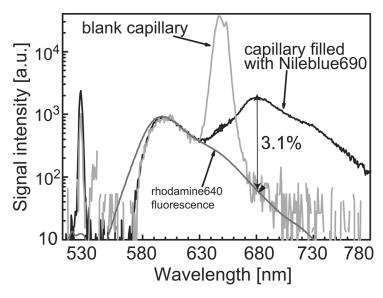
134/[416] Y. Oki et al.



**FIGURE 2** Schematic of experimental setup of fluorescence on a silicacapillary mounted dye laser array. A V-groove and PMMA adhesive was mounting and refractive index matching.

absorption properties to Cy5 dye that is one of typical labeling-dyes, and its absorption cross-section is  $1.9\times10^{-16} \rm cm^2$  at 650 nm that is almost twice of that of the Cy5. The wavelength of DFB laser output was tuned at 650 nm and fluorescence from Nileblue690 solution (black line) was obtained clearly over 630  $\sim$  780 nm. The Nileblue690's concentration is 6.7 mM that corresponds to the density of  $4\times10^{-18} \rm cm^{-3}$  and the absorption length of  $27\,\mu m$ . Since the strong scattered background from pumping laser beam was observed at 532 nm it will requires a reduction of a scattered pumping beam not absorbed by the laser waveguide. To distinguish net signal and laser background, the laser scatter was also measured by using a blank capillary (light gray lines). The Nileblue690 molecules totally absorbed the beam from the DFB laser, and it reduced the intensity of the scattered laser beam as low as 0.016% of the blank sample.

However, the ASE suppression of DFB laser output spectrum still seems insufficient in wavelength region longer than 650 nm. The suppression ratio was almost 20 dB at wavelength of 680 nm, and the scattered ASE at 680 nm was as large as 3% of the signal intensity. It can estimate the detection limit of 209  $\mu$ M in concentration of the Nileblue690 approximately. It corresponds to the density of 1.26  $\times$  10<sup>-17</sup>cm<sup>-3</sup>. Therefore, it was found that improvements in the ASE suppression and scattering reduction will be needed to detect Cy5 molecules in the concentration of several  $\mu$ M. We have not optimized the



**FIGURE 3** Spectrum of the fluorescence signal from Nile Blue solution in the capillary pumped with Rhodamine640 doped DFB laser. Light gray line represents the scattered laser output with blank sample, dark gray represents the fluorescence of Rhodamine640, and black lines represents the fluorescence signal.

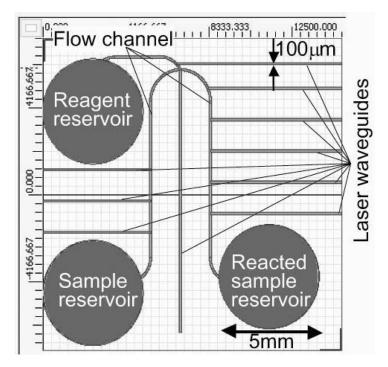
experimental condition, yet, because of the restriction of our fabrication technique, such as the alignment between the waveguide's end and the capillary position. It didn't have an acceptable accuracy and reproducibility for an optimizing experiment. Therefore, we changed the fabrication method to the lithography process.

### 4. LITHOGRAPHY BASED FABRICATION OF FLOWCYTOMETRY CHIP

#### 4.1. Fabrication Method I

Figure 4 shows a designed mask pattern for the microflowcytometry chip using deep-UV lithography with a KrF excimer laser (Lambda Physik, COMPeX 110, 248 nm). It is superimposed image of the layers of laser-waveguide and flow-channel. Three circle-reservoirs are for sample, reagent, and mixed product. They were connected by the flow channel of  $100\,\mu m$  width. DFB laser-waveguides were located to be coupled to the flow-channel directly. Three waveguides were assumed for the sample, and five were for the mixed product, for instance. The

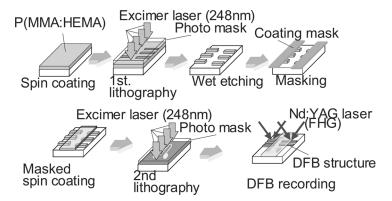
136/[418] Y. Oki et al.



**FIGURE 4** Mask design for the flowcytometry chip. The reservoirs and flow-channel are on a first layer, the laser waveguides are on a second layer.

masks were made of Cr-coated quartz substrate (ULCOAT, DUFQ-3006(S)-L, Q.T.:20, E.T.:53 sec(20°C) because of the excimer laser exposing, and they were fabricated using electron beam exposing with JBX-5000SL (JOEL, resist is OEBR-100).

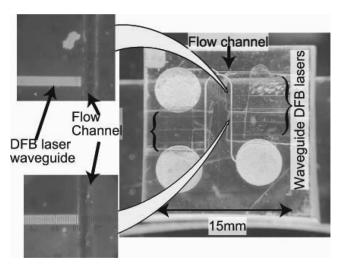
Figure 5 shows the fabrication procedure. Laser waveguide array was fabricated at first [13] with the mask for laser-waveguide. Subsequently, we used a mask-spin-coating technique to coat a P(MMA: HEMA) film partly to fabricate flow-channels, by using a film mask of 100  $\mu m$  thick. The refractive index of the P(MMA:HEMA) was monitored by a prism-coupler (Metricon, Model 2010) and was matched with the laser waveguide to minimize a scattering of the laser output. The second lithography adopted an excimer laser exposing with fluence of 10 times of the first lithography. The new mask of the flow channel was used here. The developed flow-channel has a depth of  $50 \sim 100 \, \mu m$ . Bottom schematics in Figure 5 shows their section profiles. The waveguide's output end was coupled directly to the target in the flow channel. Finally DFBs were recorded on the laser



**FIGURE 5** Fabrication procedure of the flowcytometry chip using excimer laser lithography and masked spincoating.

waveguides. In followings, we adopt the masked-spin-coating to fabricate a multiple color DFB lasers also.

Figure 6 shows a photo image of the developed chip. The flow channel looks as white lines, and DFB laser waveguides looks as red and orange lines. The left small photo images are magnified images of the couplings of the waveguide's end and flow-channel under an UV fluorescent lamp. Laser waveguide width was about  $80\,\mu m$ , and flow



**FIGURE 6** Photo image of the flowcytometry chip fabricated with procedure shown in Figure 5.

138/[420] Y. Oki et al.

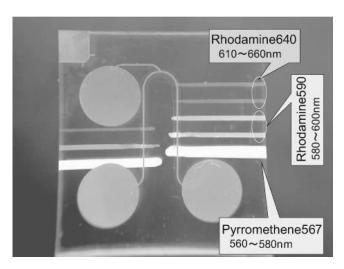
channel was  $100\,\mu m$  width. We could confirm that the output ends were coupled directly to the flow channel, and the accuracy and reproducibility were acceptable. However, there remained several problems to be solved such as:

- The masked-spin-coating made burrs at the edge of the mask. Since their height was several hundred μm, it made the covering of the flow channel difficult.
- The masked-spin-coating was also applied to integrate multi-color DFB laser waveguides, but the burrs were remained after lithography. Furthermore, masking accuracy was insufficient.

Since the covering of the flow-channel was needed to flow the sample, we tried another fabrication method.

#### 4.2. Fabrication Method II

To solve the above, we adopt novel partly coating method using a dispenser. By controlling dispenser's motion and dispensing rate, fine drawing can be performed and it made partly coating possible. This method can control the coating thickness around  $1\sim 5\,\mu m$ , and resolution of  $100\,\mu m$ . So, fine patterning will be attained by combining with the excimer laser lithography. At first, we replace the spin-coating of the dye-doped P(MMA:HEMA) in Figure 5 with the drawing.



**FIGURE 7** Photo image of the flowcytometry chip fabricated with procedure using drawn film.

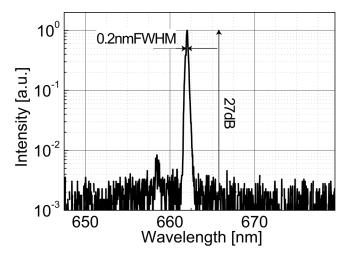


FIGURE 8 Spectrum of the DFB laser output based on the drawn film.

Secondly, we coat the P(MMA:HEMA) none-doped along to the flow-channel instead of the masked-spin-coating in Figure 5. Figure 7 shows the photo image of multiple color laser films on the chip. Three different laser dyes, such as Pyromethene567, Rhodamine590 and Rhodamine640 can cover the wavelength region of  $560 \sim 580$ ,  $580 \sim 600$  and  $610 \sim 660$  nm, respectively.

Though the lithography for laser waveguide has not been applied, the coated films must work as a planer waveguide DFB lasers. The surface quality of the coated films was evaluated by recording the DFB. Figure 8 shows an example of the DFB laser planar waveguide of the partly coating in Figure 7. The output spectrum shows the bandwidth of 0.2 nm FWHM, and it is almost similar to the traditional our DFB laser made from the spin-coated film. So the surface quality of the film is comparable to the spin-coating. Furthermore, the ASE suppression of the laser is 27 dB approximately. So, it can expect an improvement in the detection of the Nile Blue or Cy5 dye in the flow channel.

We could not find burrs like a previous chip as shown in Figure 6 in the last fabrication. So, the covering of the flow-channel seemed easy on the chip. Now we are trying to cover the flow-channel with PMMA film heated.

#### 5. CONCLUSIONS

We report about our first trial of the DFB film laser integrations on a plastic flowcytometry chip. Capillary mounting on a film laser array,

140/[422] Y. Oki et al.

film drawing technique with dispenser, and film lithography fabrication with excimer laser were tried and combined. In result, the drawn coated film and flow-channel made by the lithography could make the laser integrated flowcytometry chip successfully.

#### REFERENCES

- [1] Duarte, F. (1995). Opt. Commun., 117, 480.
- [2] Costela, A., Gar´cıa-Moreno, I., Sastre, R., Coutts, D. W., & Webb, C. (2001). Appl. Phys. Lett., 79, 452.
- [3] Maslyukov, A., Sokolov, S., Kaivola, M., Nyholm, K., & Popov, S. (1995). Appl. Opt., 34, 1516.
- [4] Sriram, S., Jackson, H., & Boyd, J. (1980). Appl. Phys. Lett., 36, 721.
- [5] Zhu, X.-L., Lam, S.-K., & Lo, D. (2000). Appl. Opt., 39, 3104.
- [6] Fukuda, M. & Mito, K. (2000). Jpn. J. Appl. Phys., 39, 5859.
- [7] Wadsworth, W. J., McKinnie, I. T., Woolhouse, A. D., & Haskell, T. G. (1999). Appl. Phys. B, 69, 163.
- [8] Ichikawa, M., Tanaka, Y., Suganuma, N., Koyama, T., & Taniguchi, Y. (2001). Jpn. J. Appl. Phys. Lett., 40, 799.
- [9] Watanabe, H., Oki, Y., Maeda, M., & Omatsu, T. (2005). Appl. Phys. Lett., 86, 151123.
- [10] Oki, Y., Aso, K., Zuo, D., Vasa, N. J., & Maeda, M. (2002). Jpn. J. Appl. Phys., 41, 6370.
- [11] Oki, Y., Miyamoto, S., Tanaka, M., Zuo, D., & Maeda, M. (2002). Opt. Commun., 214, 277.
- [12] Oki, Y., Tanaka, M., Ogawa, Y., Watanabe, H., & Maeda, M. (2006). IEEE J. Quantum Elect., 42, 389.
- [13] Oki, Y., Maeda, M., & Tanaka, M. (2004). Molecular Crystals and Liquid Crystals, 424, 55.