Research Article Vol. 28, No. 21 / 12 October 2020 / Optics Express 31729
Optics EXPRESS

Birefringent optofluidic gratings

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Abstract: A set of parallel microfluidic channels behaving as a diffraction grating operating in the Raman-Nath regime has been fabricated and studied. The diffraction efficiency of such structure can be tuned by selecting a liquid with a particular refractive index and/or optical anisotropy. Alternatively the optical properties of the liquid can be characterised by measuring the diffraction efficiency and the state of polarization of the diffracted beam. In this work, the microfluidic channels under study have been filled with penicillin molecules dissolved in water. Due to the chirality of the penicillin, the liquid has been found to have circular birefringence of 2.14×10^{-7}. The addition of the anisotropic liquid modifies the polarization properties of the microfluidic diffraction grating. The diffraction efficiency of the grating has been characterised for different probe beam wavelengths and states of polarization. Currently the diffraction efficiency of the device is low – 1.7%, but different approaches for its improvement have been discussed.

1. Introduction

Optical components made of glass, plastic and other materials like lenses, mirrors, windows, beamsplitters, prisms, gratings, polarization gratings, holograms and others show permanent characteristics, such as focal length, dispersion, diffraction efficiency and more.

In the literature attempts have been shown to fabricate permanent gratings that can be used as waveplates. For example in Ref. [1] the authors fabricated gratings by recording interference patterns with photoresist. After development a relief grating was revealed on the surface of the layer. The photoresist grating pattern with period of 300 nm was transferred to a quartz substrate by CH_3 reactive ion etching. Then by placing two gratings in tandem, quarter waveplates were obtained. Another study [2] describes the fabrication of gratings made in a photoresist. When the gratings presented a period > λ they exhibited a phase retardation and the gratings could be used as $\lambda/4$ plates. However, because they presented higher diffracted orders losses were present. On the other hand when grating periods were < λ it was possible to obtain quarter waveplates by transferring by wet etching the grating photoresist profile to an appropriate substrate with high refractive index.

Polarization gratings have been made by recording the interference pattern of two coherent beams with orthogonal polarizations linear and orthogonal. They introduce a periodic change of the state of polarization on an incident wavefront. The recording shows a birefringent spatial modulation that repeats periodically. Some authors have developed theories and experiments to explain the phenomena [3–10].

In the last 15 years a new field has been developed that is called Optofluidics [11–14] which comprises optical elements and instrumentation. Optofluidic elements are hollow pieces that can be tuned by inserting liquids with different density, refractive index and color, for example.

#404182 https://doi.org/10.1364/OE.404182
Journal © 2020 Received 30 Jul 2020; revised 7 Sep 2020; accepted 8 Sep 2020; published 6 Oct 2020

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This process let us change their optical characteristics and apply them to make instruments like refractometers, pressure meters, scanners and more [15–18]. Among the optofluidic elements it has been shown that microfluidic channels, with the form of a serpentine, behave as a grating [19]. Thus their characteristics can be changed by flowing in the channels different liquids.

Regarding the polarization gratings attempts have been carried out to fabricate them with tunable or programmable characteristics. That is, their properties when probed with polarized light can be changed at will. These polarization gratings are based on nematic liquid crystals that respond to voltage. Thus they allow for voltage-controlled waveplates to be fabricated.

In Refs. [20] and [21] polarization gratings working as beamsplitters are demonstrated. The authors report on the use of a commercial liquid crystal spatial light modulator. Each pixel of the LCSLM acts as an electrically controllable wave plate with a voltage dependent phase shift. By using different polarizations states of the probe beam it is possible to distribute the light in the zero and/or first order. These orders can present linear or circular polarizations. The second Ref. [21] describes the fabrication of a polarization beamsplitter based on binary phase gratings. The liquid crystal cell had a thickness ranging from 5 microns to 10 microns. The grating period was 20 microns. The authors vary the voltage from 0 to 5 volts in order to tune the properties of the polarization grating. The effect of the probe beam wavelength was also considered when the performance at several wavelengths was considered (488 nm, 544 nm and 632.8 nm)

Reference [22] reports on the fabrication of polarization gratings by utilizing liquid crystal cells patterned by an Atomic Force Microscope (AFM) stylus. The tunable parameters are cell thickness, pixels size and birefringence. Gratings that simulate square and blazed profiles were demonstrated. Unfortunately the use of high voltages and a cumbersome fabrication process including a lengthily processing time with the AFM stylus make these devices impractical.

Another approach to fabricate polarization gratings is reported in Refs. [23] and [24]. A mixture of a Nematic Liquid Crystal (NLC) and methylene red was used. Unlike in the previously described attempt [22], in this case the grating properties were controlled by light. A grating was first fabricated by direct electron beam lithography on SU8 and then the relief pattern was transferred to PDMS by cast molding. The gratings period was 2 microns with a thickness of about 1.5 microns. The grating area was 2 mm x 1 mm. Due to its characteristics the grating operated in the Raman-Nath regime. The mixture methylene red-NLC was poured above the PDMS grating and on top of it a glass plate was placed. This plate was coated with a thin polyamide film to induce planar alignment. Study of the intensity of the first diffracted order as a function of the polarization state of the illuminating light revealed that the intensity varied from a minimum to a maximum indicating that the illuminating light experienced a variation due to a birefringence of the NLC. Diffraction efficiencies of 35% were achieved.

More recently it was shown that with microfluidic gratings it is possible to measure liquid's refractive index. The gratings sensitivity is a function of the relief depth [25]. The liquids used so far in these microfluidic gratings were isotropic for example glycerin, thiodiethanol (TDE) and others. Now we show that microfluidic gratings that carry birefringent liquids also behave like diffraction gratings. Through experiments we study the characteristics of these new type of optofluidic gratings.

Section 2 shows the materials and method utilized to fabricate the optofluidic gratings. In Section 3 we present results from the characterization of the anisotropic fluidic mixture. Section 4 shows a characterization study of the polarization characteristics and diffraction efficiency of the diffracted orders.

2. Grating material and fabrication method

The compound optofluidic grating that is presented here consists of two sets of gratings, Fig. 1. First, there is a set of parallel relief slits that have a width (w) and a height (h), set A. Second, between the relief slits there is a second set of slits, each with a width (i), set B. This second set

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of slits carry the birefringent or optically active medium. The period of the grating is p = i + w. The dimensions of the channels were: w = 200 µm, i = 200 µm, h = 500 µm and length 5 mm. Thus the period of the grating p=400 µm. The two sets of slits transmit light. When the light is sent to the compound microfluidic grating it is diffracted and several orders will appear. The depth or height (h) was chosen large, 500 µm, because the rotation angle of the linearly polarized light is a function of the optical path length, this is explained in the following section. Thus, in the far field we have the interference of two sets of light, one passes through the first set (A) of relief slits, where the angle of the linearly plane polarized light do not experience any change, and the light that passes through the secondary grating (set B) where the linearly plane polarized light experiences a rotation due to the optically active medium. In what follows the compound optofluidic grating will be called optofluidic grating.

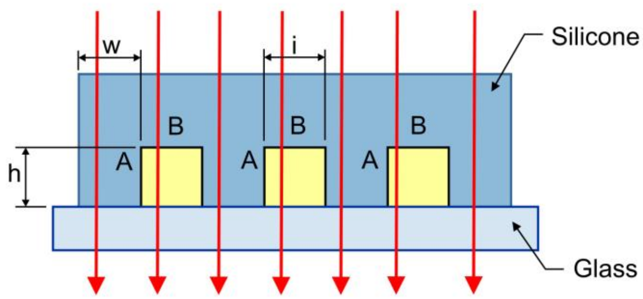

Fig. 1. Diagram showing a section of the optofluidic grating that is composed of two gratings. The set A are the relief slits and the set B are the shallow slits. Through this last set flows the optically active medium. Both sets transmit light.

The material to make the optofluidic grating was silicone (PDMS) which, among other good characteristics, is hydrophobic, shows good transmission to visible light, low wall's roughness and scattering, presents no shrinkage, it is readily available, and is easy to handle. To fabricate the microfluidic channels the soft lithographic method [25] was followed. Briefly, a silicon wafer is coated with a photoresist and soft baked on a hot plate. Next, the wafer is exposed with a micropattern generator (µPG 101, Heidelberg Instruments, Germany) with its respective CAD design, followed by a post-thermal treatment. The wafer is developed using PGMEA (484431 sigma aldich, USA), and the molds are thermally treated at 135 ^{o}C for 2 h. Then a mixture of the PDMS base and curing agent in a 10:1 ratio was prepared. The PDMS mixture was placed in a vacuum chamber to remove the gas trapped. Next the mixture was poured over the mold and the ensemble was left for 24 h. Then the cured silicone film was detached from the master. To seal the channels a glass plate was placed over them and sealed with oxygen plasma. A photograph of the microfluidic channels is shown in Fig. 2. Regarding the diffraction regime [26] the Q value can be calculated with the formula Q= $(2\pi\lambda h)/(\Lambda^2 n_0)$ where λ is the vacuum wavelength, Λ the grating spacing, n_0 the mean refractive index. The other signs have been described before. Thus, Q = 0.008. This means the grating operates in the Raman-Nath regime. By using the Raman-Nath theory it was possible to calculate the expected diffraction efficiency of the grating before filling the channels with any liquid by taking into account the depth of the grooves, the periodicity of the grating and the refractive index of the PDMS – 1.43. The theoretical value for the diffraction efficiency in the +1 order of diffraction for 633 nm was estimated to be 14.5%, while the measured diffraction efficiency was 6.3%. This reveals that there are significant losses due to scattering and reflections from the front surface of the layer containing the microfluidic structure.

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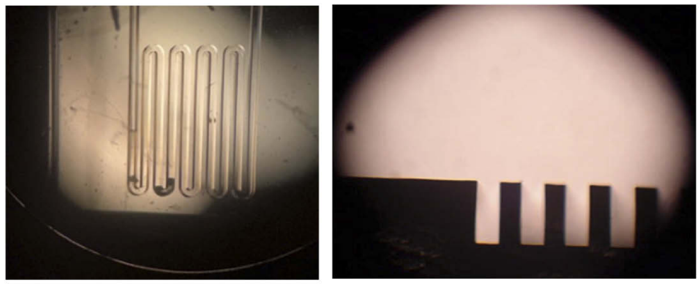

a) b)

Fig. 2. a) An optofluidic grating. b) The channels width is 200 µm, the distance between them is also 200 µm, the depth is of 500 µm. Channels were 5 mm long. The channels presented a square profile.

3. Penicillin mixture studies

Regarding the material to make the birefringent liquid it should present several characteristics. The most important one is the Specific Rotation [27] : $[\alpha]_\lambda{}^T = \alpha/(l \text{ x } \rho)$ where α is the measured rotation of the plane of polarization of a linearly polarized light in degrees, l is the optical path length and ρ is the density of the liquid, in g/ml, for a sample at temperature T (given in Celsius degrees) and illuminated with light having a wavelength λ (in nanometers). If the wavelength of the light is the yellow sodium D line, 589 nm, the symbol D is used. The sign of the rotation (+ or -) is always given. It is common that the optical rotation decreases approximately when the wavelength (λ) of light increases ($\alpha = k/\lambda^2$, Law of Inverse Squared) where k is a constant. In some cases α increases when the light wavelength also increases, then changes its direction to cross zero line and changes sign [28]. This behavior is called anomalous optical rotatory dispersion and is given by Drude's equation $\alpha = k/(\lambda^2 - \lambda_0{}^2)$ where λ_0 is the wavelength of an electronic transition. Other characteristics that the birefringent liquid should present is being transparent to visible light, present low density, etc. This last characteristic should be fulfilled to avoid clogging of the microchannels.

The chemical powder that was chosen in this study was penicillin G which has a specific rotation of $[\alpha]_D{}^{20} = +290 \pm 5^o$ (2% in H_2O). A mixture with large specific rotation should be used because the optical path, or depth of the grating relief, is relatively small (500 µm). To study the optical characteristics of the penicillin-water mixture samples with 1.2 g of penicillin and 1.5 ml of water were prepared. They were magnetically stirred until the penicillin was fully dissolved.

As previously stated the optical rotation Law of Inverse Squared ($\alpha = k/\lambda^2$) is approximately followed by the anisotropic mixtures. To find out the specific rotation behavior of the mixture a polarimeter was used. Three lasers (632.8 nm, 543 nm, 468 nm), a LED (405 nm) and a sodium lamp (589 nm) were used to study the spectral dependence of the optical rotation. The mixture was poured in a glass cell (5 mm thickness). The result is shown in the Fig. 3(a). It is noticed that for short wavelengths the rotation angle is larger than for long wavelengths. Another experiment to characterize the mixture comprised the behavior of the rotation angle as a function of the optical path. Several glass cells with different thicknesses were made. They were placed in the polarimeter. The same ratio of penicillin/water was poured in each cell, linearly polarized red light (632 nm) was used. The cells were placed in the beam path, one at a time, and the analyzer was rotated until zero intensity was achieved. The following graph shows the results, Fig. 3(b).

Based on the plot of Fig. 3(b) we can calculate the circular birefringence, the difference of refractive indices for circularly polarized light which rotates anticlockwise (n_A) and clockwise (n_C), (n_A-n_C). The Rotation of the plane of vibration is given by the following relation [29–30] $(\alpha) = (\pi d/\lambda)(n_A - n_C)$; α in radians, d is the distance traveled in the medium, by algebra we can

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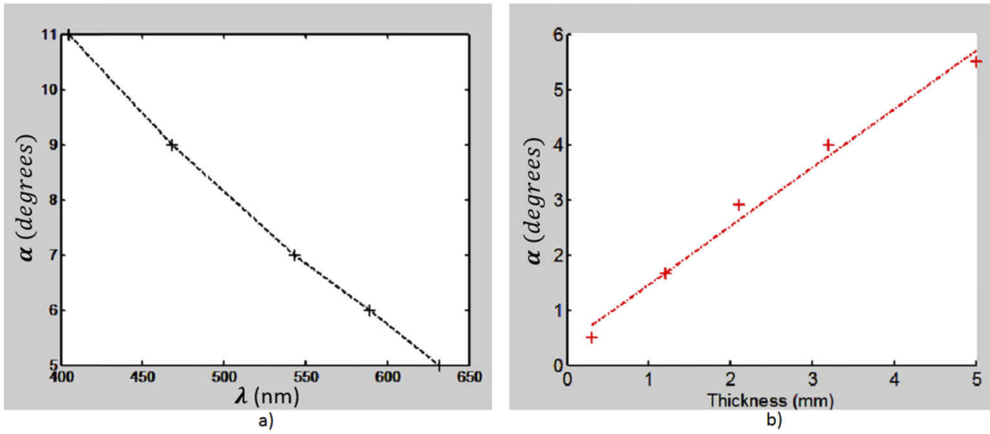

Fig. 3. α (degrees) as a function of a) λ (nm) and b) cell thickness (mm). A linear fit is also showed.

obtain (n_A-n_C)= αλ/πd (λ in Angstroms). By taking the linear fit we got a value for the slope and then we calculated the (n_A-n_C) value and got 2.14×10^{-7}. By comparison the (n_A-n_C) of a 1 mm thickness quartz plate is 7.1×10^{-5}. Usually the rotation produced by a liquid is considerably less than that produced by a crystal. For example 10 cm of turpentine rotates sodium light –37^o whereas an equal thickness of quartz would rotate it at an angle almost two orders of magnitude larger +2172^o [29].

Another experiment was carried out to find the behavior of the mixture when white light from a frosted bulb was used. The mixture was poured in a glass cell having 1.2 mm thickness. Then the cell was placed in a polarimeter. The analyzer was rotated until no light was transmitted, Fig. 4(a). Then the analyzer was rotated about 2^o in the anti-clockwise direction and the color showed in Fig. 4(c) was observed. If the analyzer was rotated in the clockwise direction by 1.5^o the color in the Fig. 4(e) was observed. By investigating the images with the computer program "Color Model RGB" [31], the plots in Fig. 4(b), Fig. 4(d) and Fig. 4(f) were obtained. Here the ordinate axis is the intensity level that was captured by the camera sensor. The intensity levels in each channel R, G, B are in a range from 0 to 255. We can see that the color in Fig. 4(d) is mainly a mixture of red and green. Blue color is faint. However, Fig. 4(f) shows that the mixture of green and blue colors is present, with blue the stronger color.

Because the studies were related with polarized light it was necessary to investigate if the microfluidic channels (serpentine) presented strains that could give a birefringence. Thus, using white light, we placed the serpentine, without any liquid, in the polarimeter and no traces of birefringence were found. To investigate the behavior of the penicillin/water mixture in the microfluidic channels the mixture was injected in the serpentine and this was placed in the polarimeter. The photographs in Figs. 5(a) and 5(c) show three of the channels. One channel was chosen to make the color measurements. In 5(a) the analyzer was rotated in the anti-clockwise direction and in 5(c) in the clockwise direction. Also are shown the graphs of the colors in 5(b) and 5(d).

The transmittance of the mixture penicillin/water for just one wavelength also was investigated. A LED (λ = 405 nm) was used as the light source. The microfluidic system, injected with the mixture, was placed in the polarimeter with crossed polarizers, Fig. 6. There were two displays depending on the analyzer angular position: a) at crossed position the background was seen dark and the channels were bright, b) when the analyzer was rotated about 1^o the channels became dark and the background bright. Display a) was present because linearly polarized light passing through the silicone, where no channels were present, did not experienced any optical rotation and

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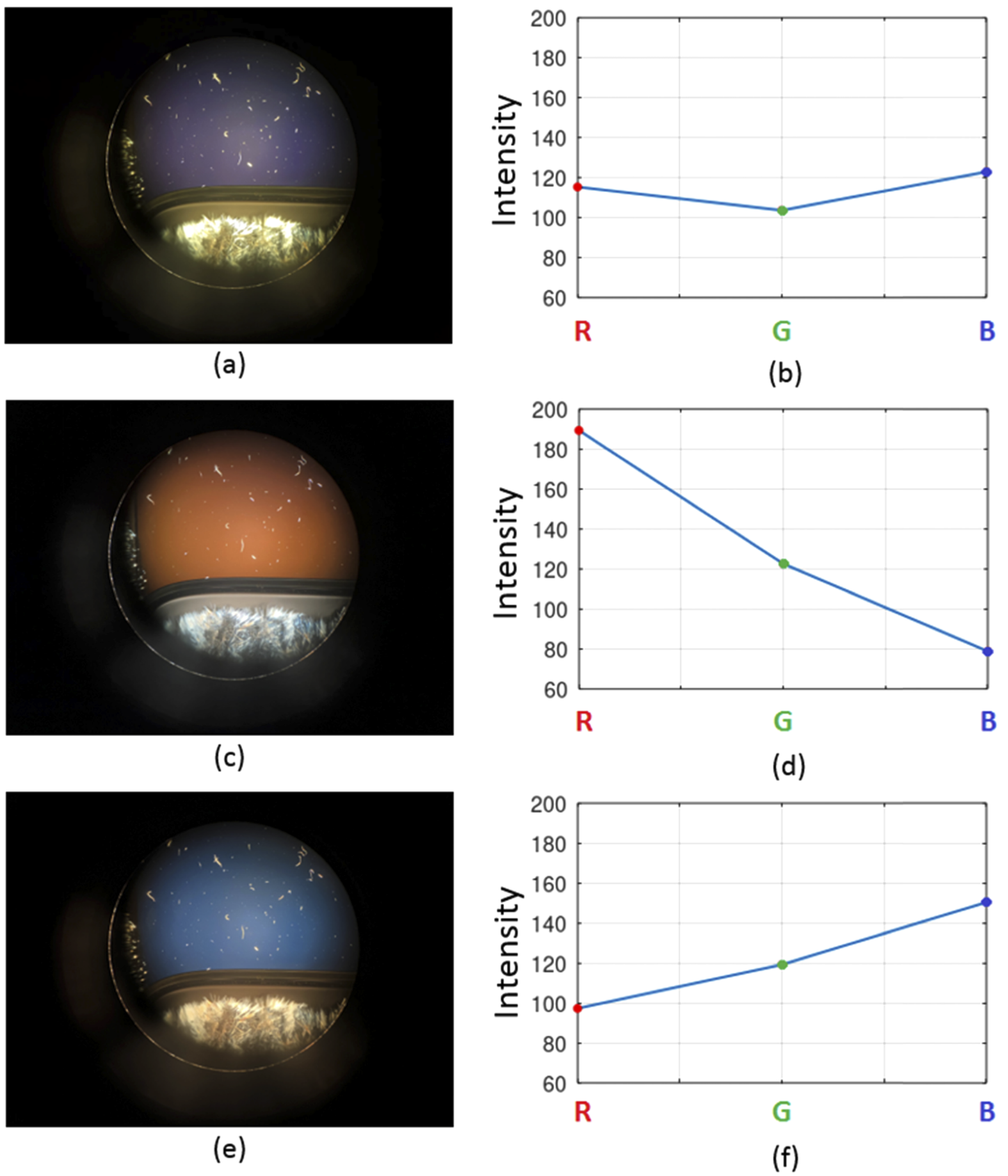

Fig. 4. These photographs show the transmittance of a cell where a mixture of 1.2 g penicillin/ 1.5 ml of water was poured in a 1.2 mm thick cuvette. A frosted bulb was used as a light source. In a) the polarizer and analyzer were closed (Note. The photograph shows a light purple color because the camera gave a large time of exposure. However, with the naked eye no light was seen). In c) the analyzer was rotated in the anti-clockwise direction by about 2^{o} and in e) the analyzer was rotated in the clockwise direction by about 1.5 degrees. Plots in b), d) and f) show the mean intensity of the RGB components of a small central region from each image on their left.

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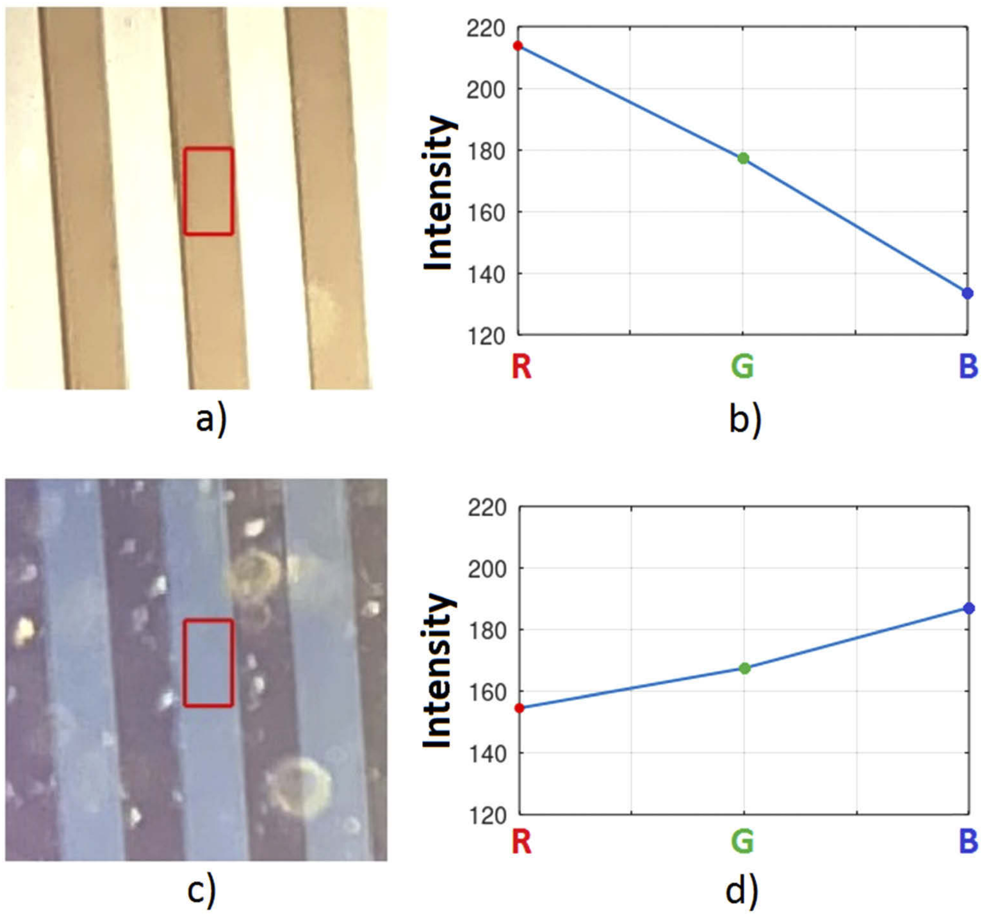

Fig. 5. The serpentine with a mixture of penicillin-water was placed between crossed polarizers. A white lamp was used as a light source. Photographs in a) and c) show the same microchannel. The red rectangles show the studied area. In a) the analyzer was rotated about 2^{o} in the anti-clockwise direction. In c) it was rotated 1.5^{o} in the clockwise direction. The colors intensity were studied with a computer program and are shown in b) and d).

the analyzer stopped the light. However, light that passed through the microchannels experienced a rotation. b) when the analyzer was rotated it stopped the light that suffered a rotation when passed through the microchannels and let pass light that passed through the silicone where no channels were present. This change of contrast has been mentioned in Ref. [32] as a means to change the contrast reversal of a transparency.

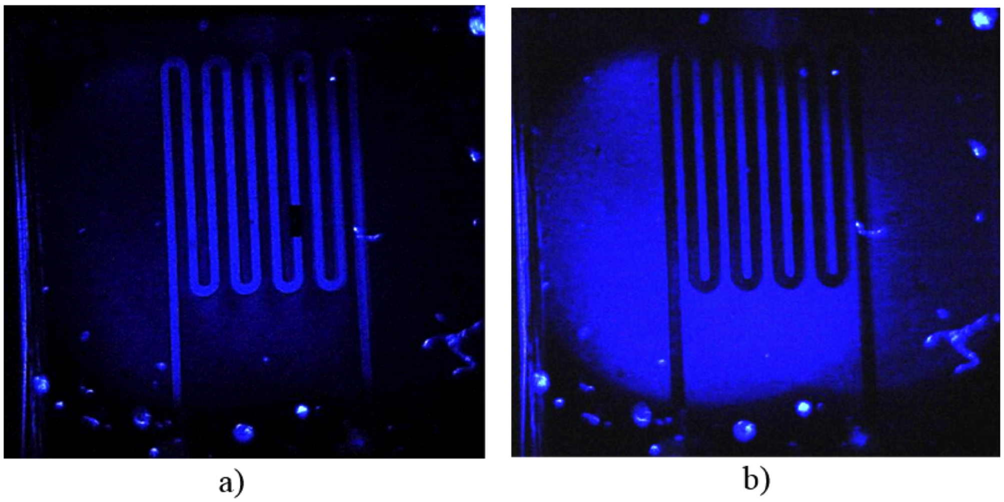

Fig. 6. The optofluidic grating, with the penicillin/water mixture, was inserted in a polarimeter with crossed polarizers. A LED was used as the light source ($\lambda = 405$ nm). The image seen in a) was seen. The microchannels were bright. The opposite was the case when the analyzer was rotated by about 1^{o}, b).

4. Polarization behavior of the diffracted light

Several experiments were carried out to characterize the polarization behavior of the diffracted orders given by the optofluidic grating filled with the optically active mixture penicillin/water (1.2 g penicillin in 1.5 ml of distilled water). A scheme of the optical configuration is shown in Fig. 7. Three light sources providing linearly polarized light were used, one at a time: two stabilized He-Ne lasers emitting red ($\lambda = 632.8$ nm) and green light ($\lambda = 543$ nm), and a laser emitting blue light ($\lambda = 468$ nm, Diode Pumped Solid State Laser, DPSSL, Neodymium-doped YAG Crystal). The three wavelengths were used because the Specific Rotation is a function of the wavelength. Temperature was stable within $\pm$ 2 ^{o}C and was at the room temperature value.

The $\lambda/2$ plate was used to rotate the plane of linearly polarized light. Its angle is called θ and had two positions 0^{o} and 45^{o}. 0^{o} means the linearly polarized light was parallel to the microchannels longest dimension. Analyzer angle ϕ was rotated in steps from 0^{o} to 360^{o}. Polar plots were used to better understand the behavior of first order light polarization. Water was the first liquid injected in the serpentine. It does not show optical activity. These results will be compared with the ones when an optical active mixture is used (Penicillin). Plots in Fig. 8 show the results when the $\lambda/2$ plate was rotated θ: 0^{o} and 45^{o} and the analyzer rotated from ϕ: 0^{o} to 360^{o}. Three wavelengths were used. The radial coordinate is the first order intensity.

We can see that for the three wavelengths considered and the value of $\theta = 0^{o}$ the first order polarization behavior is similar. It is maximum when the analyzer angle ϕ has the values of 0^{o} and 180^{o} and minimum when ϕ is 90^{o} and 270^{o} meaning the first order polarization is linear. This behavior is also shown when the $\lambda/2$ plate was rotated by θ=45^{o}.

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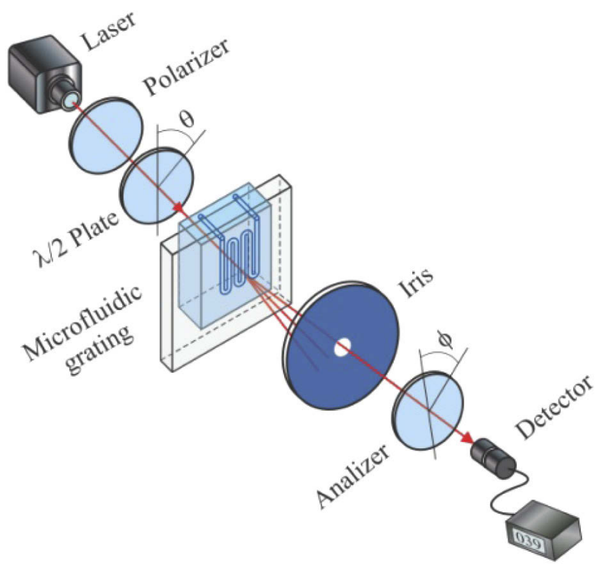

Fig. 7. Scheme of the optical configuration used to study the first diffracted order behavior when the microfluidic grating had isotropic or anisotropic liquid in its channels.

To test the microfluidic grating behavior when an anisotropic mixture was injected in the channels a mixture of 1.2 g penicillin/ 1.5 ml of water was prepared and introduced in the channels, blue light was used because with this wavelength specific rotation is greater (Fig. 3). Results are shown in Fig. 9. We can notice that they are similar to those plots shown in Fig. 8.

A note on the mixture behavior when it is illuminated with polarized light is made now. It can be noticed in the plot of Fig. 9 b) that the first and last measurements are not the same as it should. These measurements correspond to the angles 0^o and 360^o. We have noticed that when the mixture is under light illumination the intensity measurements fluctuate even when illuminating power is in the order of a few μW. We have kept fixed the parameters that affect the Specific Rotation like temperature, wavelength, light intensity and polarization. Thus, possibly the mixture absorption/scattering increases during the illumination. Studies reported previously [33–35] show that degradation of antibiotics (like penicillin) can be achieved with visible light. Besides, penicillin removal from waste water has been achieved by the (UV/ZnO) photocatalytic method using ZnO nanoparticles and UV light, and also using the nanocomposite Ag-AgBr/TiO_2/RGO, this time using white light emitted by diodes.

We have observed above that when illuminating light has linear polarization the first order polarization also was linear (Fig. 8 and Fig. 9) even when different wavelengths were used and two θ angles were considered ($\theta = 0^o$ or 45^o). Besides this study, first order polarization state was studied when illuminating light had circular polarization. To achieve this a λ/4 plate was inserted in the illuminating beam path. Again the analyzer was rotated in steps from 0^o to 360^o. Results are shown in Fig. 10. It is possible to notice that first order beam is circularly polarized.

An important parameter of diffraction gratings is their diffraction efficiency. To know the behavior of this parameter when illuminating light changed from linear to elliptical to circular a λ/4 plate was inserted in the illuminating beam path and rotated by steps from 0^o to 45^o. First order intensity was measured. Results can be found in Fig. 11. We can notice that the diffraction efficiency remains constant even when the reading light state of polarization changed. Diffraction Efficiency value was about 1.7%.

Several challenges were noticed in relation to the mixture stability. When the mixture was poured in the glass cells some strains appeared in the bulk of the mixture after some 20 minutes.

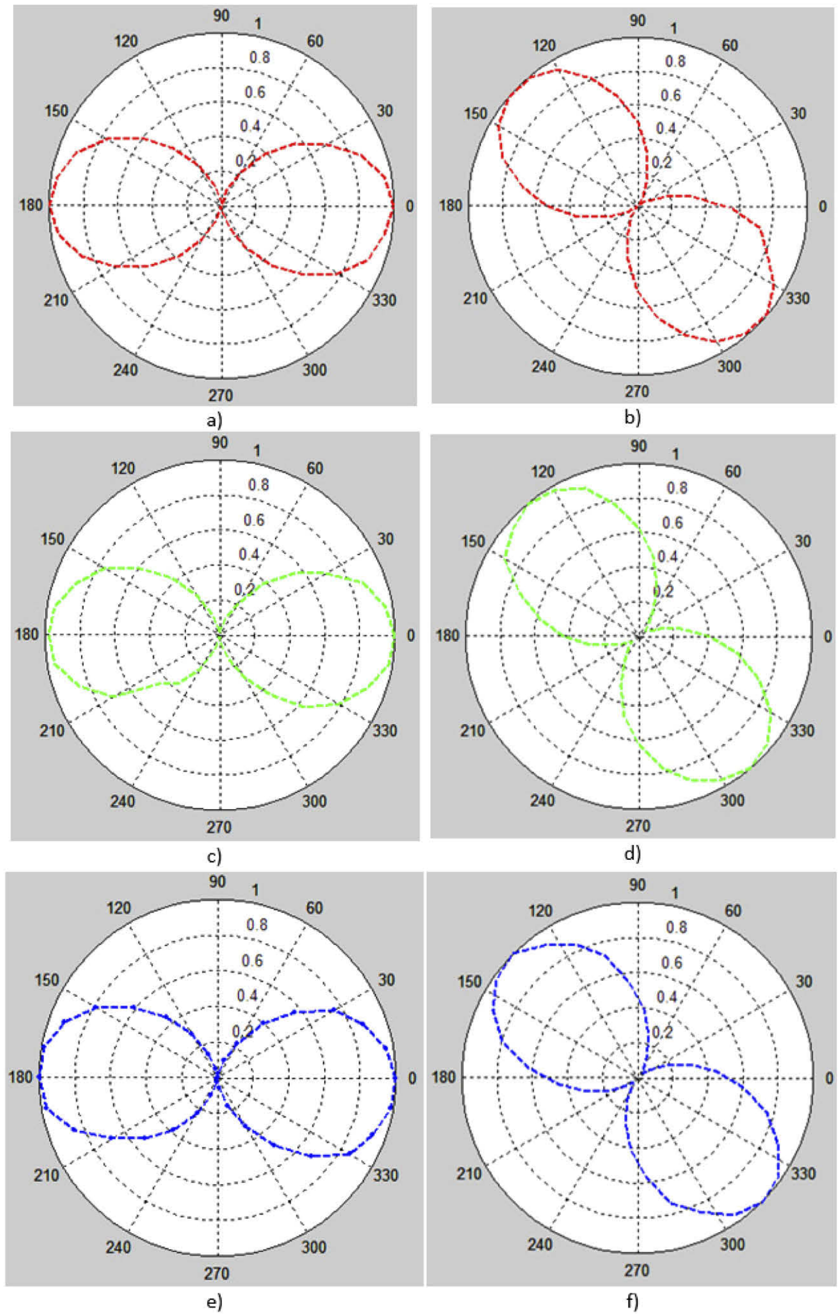

Fig. 8. Polar plots showing the normalized first order intensity behavior (radial coordinate) as a function of the analyzer angle ϕ when a microfluidic grating with water was investigated. Three wavelengths were used, 632.8 nm, (Fig. a) and Fig. b)), 543 nm (Fig. c) and Fig. d)), and 468 nm (Fig. e) and Fig. f)). In Fig. a), Fig. c) and Fig. e) illuminating linearly polarized light angle was $\theta=0^{o}$ and in Fig. b), Fig. d) and Fig. f) was 45^{o}.

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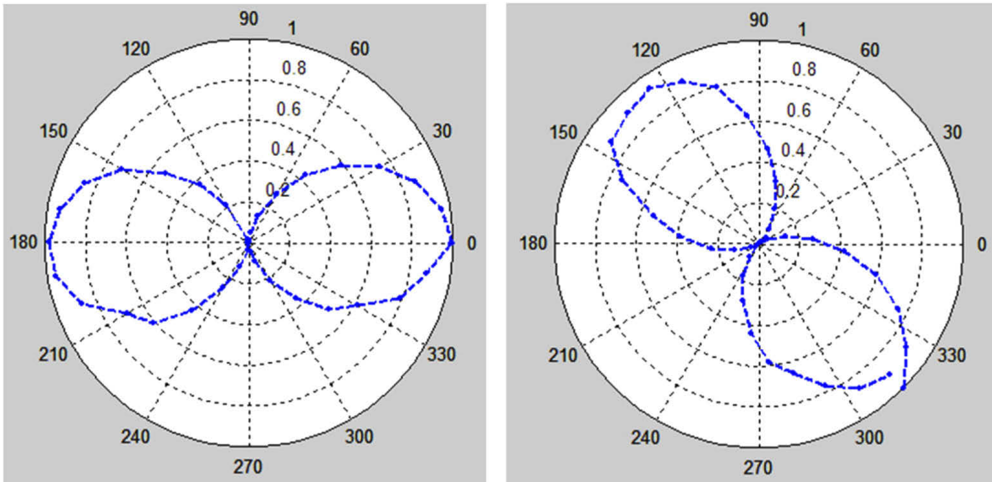

a) b)

Fig. 9. Normalized First order intensity behavior (radial coordinate) as a function of analyzer angle ϕ (0^o to 360^o) when a microfluidic grating had the mixture penicillin/ water. In a) $\lambda/2$ plate had an angle $\theta=0^o$, in b) $\theta=45^o$.Blue light was used.

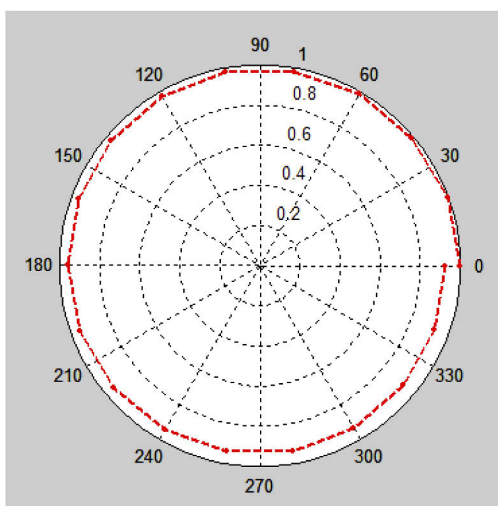

Fig. 10. First order intensity (radial coordinate) as a function of analyzer angle when a microfluidic grating had a mixture of penicillin/ water. Incoming or reading light ($\lambda = 632.8$ nm) was circularly polarized. It is possible to notice that first order also showed a circular polarization.

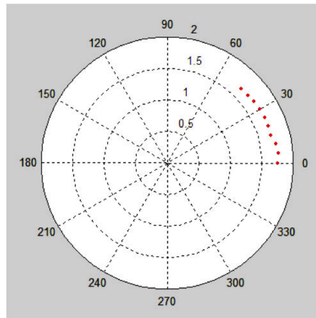

Fig. 11. Difraction Efficiency (radial coordinate) as a function of λ/4 plate angle inserted in the reading beam. The microfluidic grating had a mixture of penicillin/ water. Incoming light polarization began as linear (0^o), then passed through elliptical and at 45^o became circular. Mean diffracion efficiency was 1.7%. Red light ($\lambda = 632.8$ nm) was used.

These were not due to temperature differences and were not present when the serpentine was used. Besides that, we noticed that the mixture showed high capillarity because after some time it began rising to the upper part of the glass cells windows. Then the water began to evaporate and the penicillin, in the form of powder, was seen. An optimization of the stability of the fluid utilized in the optofluidic grating will be needed in order to avoid these negative effects.

5. Conclusions

We have presented an optofluidic grating where optically active liquids can be injected. In the fabrication method grating parameters like the width of the channels, distance between them and deep of the channels can be controlled. The isotropic liquid can be changed to show desired characteristics. All these parameters will let tune the grating for an specific purpose. Liquid crystals present desirable characteristics but unfortunately their fabrication method needs special and expensive equipment. Also for their application high voltages, electronic instrumentation and computers are needed to address the cells. In the optofluidic gratings, due to the liquid anisotropy, the incoming wavefront experiences a periodic change in its state of polarization. Studies of the optically active liquid, and polarization characteristics of the first diffracted order as a function of the wavelength, and state of polarization of the illuminating light have been carried out. Several challenges and limitations were observed, such as liquid stability and the small optical rotation (in order of 1-2 degrees) due to the short optical path (500 μm) in the microfluidic channels. It was also determined that the diffraction efficiency of the grating was relatively low – 1.7%. Such low diffraction efficiency would not be an issue if the device is used as a sensor, but is definitely an obstacle if the device is used a tunable beam splitter for example. A number of different approaches can be utilized to address the identified challenges. The diffraction efficiency can be increased by fabricating microfluidic channels with sizes (depth and width) that will allow operation in Bragg regime, with a single diffraction order. For example if the depth of the channels is kept 500 μm their width has to be reduced to 5 μm with distance between the channels of 5 μm for the grating to operate as a Bragg grating. This geometry is technically challenging, but not impossible to achieve, especially with the recent developments in 2 photon polymerization patterning. In terms of boosting the polarization properties of the optofluidic grating, this could be achieved either by selecting a liquid with larger optical rotation or increase the concentration of the chiral molecules. The second approach is less realistic because it already has been shown to cause instabilities and crystallization of the solute. Alternatively, the depth of the channels can be increased, thus leading to larger optical path and larger rotating angles for the probe beam.

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Acknowledgments

We thank Ismael Torres-Gomez, Geminiano Martinez-Ponce, Natiely Hernandez-Sebastian, Jaqueline Muro Isamar and Raymundo Mendoza.

Disclosures

The authors declare no conflicts of interest.

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