Measurement of the refractive index by using a rectangular cell with a fs-laser engraved diffraction grating inner wall

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Abstract: A very simple method to obtain the refractive index of liquids by using a rectangular glass cell and a diffraction grating engraved by fs laser ablation on the inner face of one of the walls of the cell is presented. When a laser beam impinges normally on the diffraction grating, the diffraction orders are deviated when they pass through the cell filled with the liquid to be measured. By measuring the deviation of the diffraction orders, we can determine the refractive index of the liquid.

OCIS codes: (290.3030) Index measurements; (230.1950) Diffraction gratings; (230.4000) Microstructure fabrication; (140.2020) Diode lasers; (040.1520) CCD, charge-coupled device.

References and links

1. M. De Angelis, S. De Nicola, P. Ferraro, A. Finizio, and G. Pierattini, "A reflective grating interferometer for measuring the refractive index of liquids," Pure Appl. Opt. **5**(6), 761–765 (1996).
2. S. Singh, "Diffraction method measures refractive indices of liquids," Phys. Educ. **39**(3), 235 (2004).
3. J. Warren, Smith, "Diffraction grating," in *Modern Optical Engineering*, ed. (McGraw-Hill SPIE, 2000).
4. S. Kedemburg, M. Vieweg, T. Gissibl, A. Finizio, and H. Giessen, "Linear refractive index and absorption measurement of nonlinear optical liquids in the visible and near-infrared spectral region," Opt. Mater. Express **2**, 1588–1611 (2011).
5. S. Ariponnammal, "A novel method of using refractive index as a tool for finding the adultration of oils," Res. J. Recent Sci. **17**, 77–79 (2012).

1. Introduction

Some methods to measure the refractive index of liquids using a diffraction grating combined with a rectangular cell have been reported [1,2]. In [1], the cell is divided into two parts: a part is filled with the liquid sample and the other part is empty. The refractive index is determined by the interference of the + 1 and −1 diffraction orders of two light beams: the beam that passes through the empty portion interferes with the beam that passes with the sample liquid. This technique requires knowledge of the width of the rectangular cell. In [2], the refractive index is obtained by knowing the width of the cell and by measuring the distance between the zeroth-order beam and the first-order beam, when the cell is empty and when the cell is filled with the test liquid. In this method the effect of the glass cell is ignored, so that it is an approximate technique.

In this work, we present a very simple method to obtain the refractive index of liquids by using a diffraction grating and a rectangular glass cell. In this method, the grating is directly engraved on the inner face of one wall of the cell, so that the grating is in contact with the sample liquid contained in the cell. The diffraction orders, from a laser beam impinging normally on the diffraction grating, are propagated through the test liquid and then they are deviated when crossing the back wall of the cell. The locations of the spots of these diffraction orders were recorded in two cases: when the cell is empty and when it is filled with the liquid. By measuring the vertical separation between the spot of order-m when the cell is empty and the spot of order-m when the cell is filled with the liquid we can determine the refractive index of the liquid. In this method, it is not necessary to know the refractive index and thickness of the walls of the cell.

2. Diffraction grating

A diffraction grating consists of a series of precisely ruled lines on a clear (or reflecting) base. Light can pass directly through the grating, but it is also diffracted, producing an interference pattern whose maxima are found at angles θ given by [3]

$$\sin\theta_m = \frac{m\lambda}{S} \pm \sin I, \tag{1}$$

where λ is the wavelength, I is the angle of incidence with respect to the grating normal, S is the spacing of the grating lines, m is an integer, called the order of the maximum, and the positive sign is used for a transmission grating, while the negative is used for a reflecting.

On the other hand, if λ_o is the wavelength of light in the air (n_o ~1), then the wavelength of the light in a medium of refractive index n is given by

$$\lambda = \frac{\lambda_0}{n}. \tag{2}$$

Substituting Eq. (2) into Eq. (1) gives

$$\sin\theta_m = \frac{m\lambda_0}{nS} \pm \sin I. \tag{3}$$

For a transmission grating Eq. (3) becomes

$$\sin\theta_m = \frac{m\lambda_0}{nS} + \sin I. \tag{4}$$

3. Diffractometer

Consider a diffraction grating engraved on the inner face of one of the glass walls of a rectangular cell containing the liquid with refractive index n_2 as shown in Fig. 1. The cell, placed in the air with refractive index n_1 ~1, has a wall with thickness t and refractive index n, and the separation between their glass walls is w. When a laser beam of wavelength λ_0 impinges on the diffraction grating, the maxima are produced at angles θ_m given by Eq. (4). These maxima hit the far wall at distances d_m of the zeroth-order line as shown in Fig. 2. Now, if $(\theta_1)_m$ is the angle of the maximum when the cell is empty, and $(\theta_2)_m$ is the angle of the maximum when it is filled with the liquid to be measured, then, from the geometry of the Fig. 2 we find

$$(d_i)_m = w\tan(\theta_i)_m = w\frac{\sin(\theta_i)_m}{\sqrt{1-\sin^2(\theta_i)_m}}, \tag{5}$$

where

$$\sin(\theta_i)_m = \frac{m\lambda_0}{n_i S} + \sin I, \quad i = 1,2 \text{ and } m = 1,2,3,\ldots, \tag{6}$$

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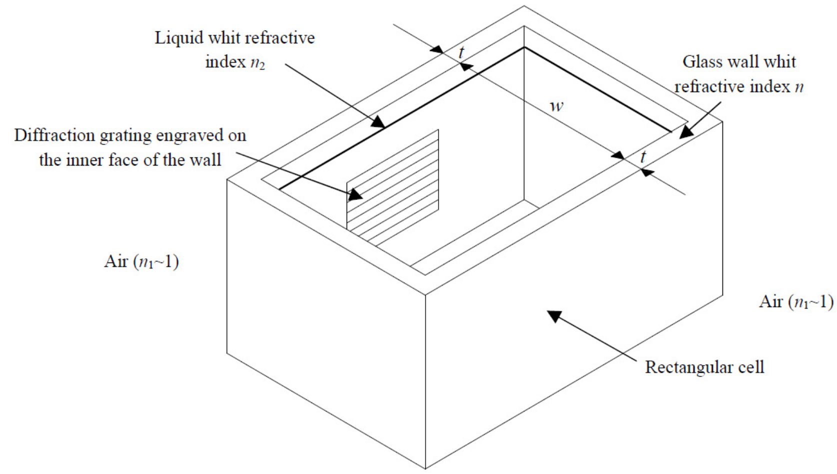

Fig. 1. Diffractometer used for measuring the refractive index of liquid substances.

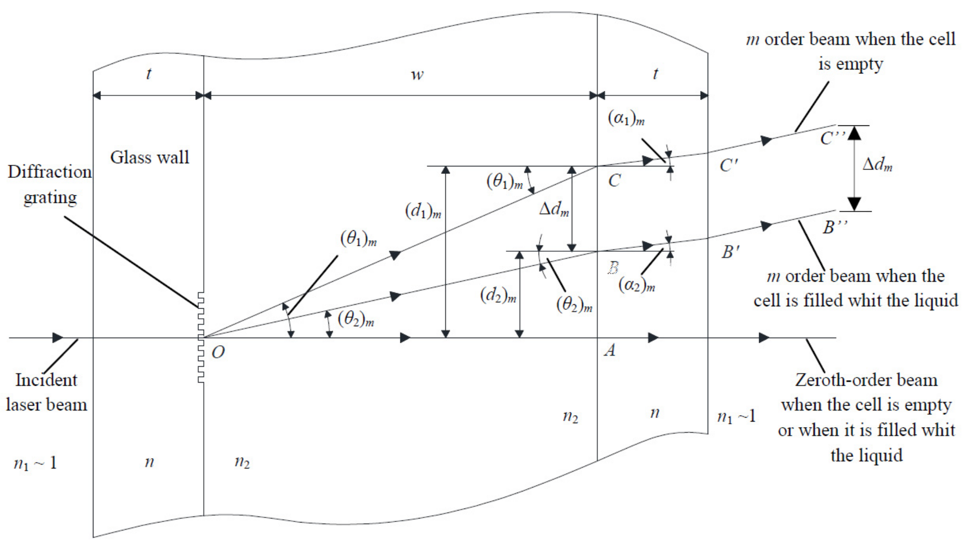

Fig. 2. Diffraction angles and paths for the m order beam in the two cases: when the cell is empty and when the cell is filled with the liquid.

In addition, if $(d_1)_m$ and $(d_2)_m$ are the distances of the maxima when the cell is empty and when it is filled whit the liquid, respectively, then the vertical separation Δd_m between these two maxima (see Fig. 2) is given by

$$\Delta d_m = (d_1)_m - (d_2)_m, \ \ m = 1, 2, 3, \ldots, \tag{7}$$

From Eqs. (5)-(7) it follows that

$$\Delta d_m = w \frac{m\lambda_0}{S} \left(\frac{1}{\sqrt{1 - \left(\dfrac{m\ \lambda_0}{S} + \sin I \right)^2}} - \frac{1}{\sqrt{n_2^2 - \left(\dfrac{m\ \lambda_0}{S} + n_2 \sin I \right)^2}} \right). \tag{8}$$

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From Eq. (8), we isolate n_2 to obtain

$$n_2 = -\frac{m\lambda_0}{S}\left(\frac{\sqrt{1+\left(\Delta d_m/w-\frac{m\lambda_0/S+\sin I}{\sqrt{1-\left(m\lambda_0/S+\sin I\right)^2}}\right)^2}}{\Delta d_m/w-\frac{m\lambda_0/S+\sin I}{\sqrt{1-\left(m\lambda_0/S+\sin I\right)^2}}+\sin I\sqrt{1+\left(\Delta d_m/w-\frac{m\lambda_0/S+\sin I}{\sqrt{1-\left(m\lambda_0/S+\sin I\right)^2}}\right)^2}}\right), \tag{9}$$

where m is the diffraction order, S is the period of the grating, λ_0 is the wavelength of the laser, I is the incidence angle with respect to the grating normal and Δd_m is measured experimentally.

Now, if the laser beam impinges normally on the diffraction grating at $I = 0°$ ($\sin I = 0$), then Eq. (6) gives

$$\sin\left(\theta_i\right)_m = \frac{m\lambda_0}{n_i S}, \quad i = 1,2 \text{ and } m = 1, 2, 3, \ldots, \tag{10}$$

and Eq. (9) takes the form

$$n_2 = \frac{m\lambda_0}{S}\sqrt{1+\frac{w^2\left(1-\frac{m^2\lambda_0^2}{S^2}\right)}{\left(w\frac{m\lambda_0}{S}-\Delta d_m\sqrt{1-\frac{m^2\lambda_0^2}{S^2}}\right)^2}}. \tag{11}$$

On the other hand, by applying the Snell's law at the inner surface of the far wall of Fig. 2 (interface liquid-glass), for the case of an empty cell it is found:

$$n_1 \sin\left(\theta_1\right)_m = n\sin\left(\alpha_1\right)_m, \tag{12}$$

and, for the case of a cell filled with the liquid:

$$n_2 \sin\left(\theta_2\right)_m = n\sin\left(\alpha_2\right)_m, \tag{13}$$

From Eqs. (10), (12) and (13) it is easy to show that $\alpha_1 = \alpha_2$; therefore the path *CC'* is parallel to path *BB´* and, thus, the output beams *C´C"* and *B'B"* are parallel with a vertical separation between them equal to Δd_m. If the laser beam impinges normal to the grating, Δd_m depends only of the refractive index n_2 of the liquid and of the diffraction order m, as can be seen from Eq. (8). Namely, Δd_m is independent of the refractive index n and thickness t of the glass walls of the cell.

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4. Procedure and experimental results

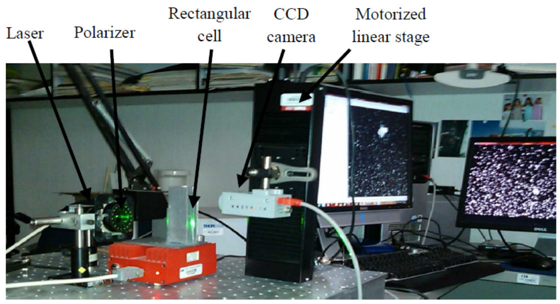

Fig. 3. Experimental arrangement used to obtain the refractive index of liquids.

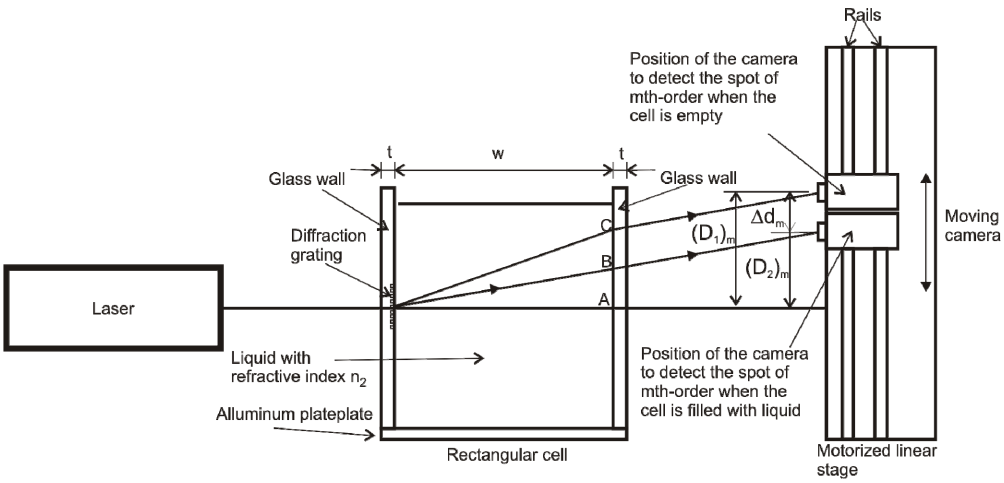

Fig. 4. Optical scheme used to detect the positions of the spots of the diffraction orders.

In the experimental configurations shown in Figs. 3 and 4, a diffraction grating of 6 μm of period was engraved on the inner face of the nearest glass wall of a rectangular cell of dimensions t = 1.03 mm, w = 34.79 mm and an inner width and height of 6 mm x 75 mm. A diode laser beam (532 nm, 5 mW), whose intensity was adjusted with a polarizer, impinges normally on the diffraction grating. The diffraction grating of 6 μm, shown in Fig. 5, was engraved by laser ablation with a Newport laser uFAB Microfabrication workstation and an amplified 50 fs Ti:Saphire laser system. The repetition rate and average power of the fs laser pulses used for the diffraction grating micromachining were 1 kHz and 300 μW, respectively. It is worth to mention that one of the advantages of engraving by laser ablation is the robustness of the grating, which, for this particular case, means that the same cell can be, and was used for all the refractive index measurements reported in this work. After each measurements the whole cell was cleaned with acetone, using tissue paper and cotton swabs, without taking any special care and no noticeable degradation on the grating performance.

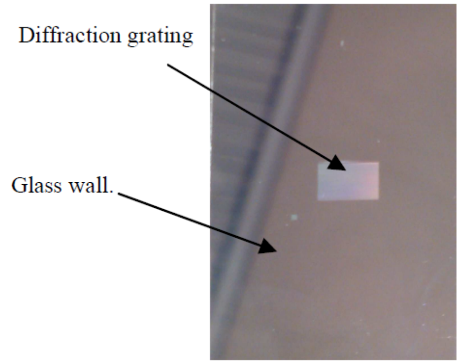

Fig. 5. Diffraction grating of 6 μm engraved on the inner face of one wall of the cell.

In Fig. 4, the exact positions of the centroids of the spots of the diffraction orders were measured using a CCD camera (Pixelink, Model PL-A741, pixel of 6.7 μm), which was placed at a distance of approximately 70 mm from of the cell and mounted on a motorized linear stage (Thorlabs, NRT150/M-150 mm, motor 90843158) in order to move it vertically. The images of the spots of first-order for the air and the water that were captured by the CCD camera are shown in Fig. 6.

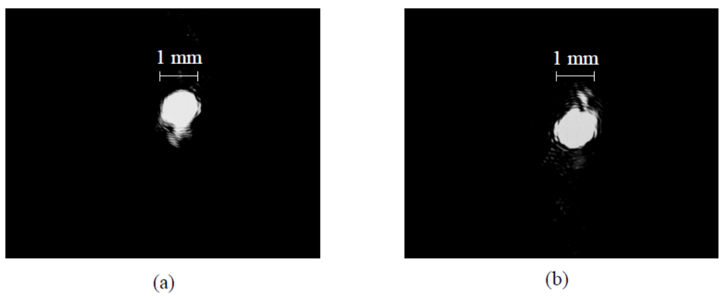

Fig. 6. Images of the spots of first-order for: (a) air and (b) water.

The images of the spots captured by the CCD camera were processed with a median filter using a 3 x 3 sampling window, and the centroids of the spots of the processed images were obtained using the mass center method. The digital image processing was performed using MATLAB software.

As shown in the same Fig. 4, if $(D_1)_m$ and $(D_2)_m$ are the positions of centroids of the spots of m order when the cell is empty and when it is filled, respectively, then

$$\Delta d_m = \left(D_1\right)_m - \left(D_2\right)_m . \tag{14}$$

For the zeroth-order and the first two diffraction orders, the laser intensity was adjusted whit the polarizer to avoid saturation in the CCD camera. In the measurement of the remaining diffraction orders, the polarizer was rotated to allow the transmission of the maximum intensity.

The laboratory has a Mini-Split air conditioner that fixes the temperature at 20 °C. Table 1 shows the measured refractive index n_2, by using Eq. (11), of the water, castor oil and ethanol along with the values of Δd_m. It can be observed that the average index values of these liquids closely agree with the reported values in [4, 5]. As shown in Table 1, only the first five diffraction orders were included, since the irradiance of the spots of the next orders was not enough to obtain their centroids with the required precision. The grating diffraction orders powers of Fig. 5 were as follows: 2.98 mW for the zeroth-order, and 158.7 μW, 13.65 μW, 6.61 μW, 2.43 μW and 1.64 μW, for the orders of the 1 to the 5, respectively. The powers

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were measured with a digital optical power meter (Thorlabs, PM100, photodiode sensor S120B).

Table1. Measured refractive indices n_2 of the water, castor oil and alcohol at 532 nm.

	Water		Castor oil		Ethanol	
m	Δd_m (mm)	n_2	Δd_m (mm)	n_2	Δd_m (mm)	n_2
1	0.7848	1.3371	1.0042	1.4767	0.8324	1.3651
2	1.6089	1.3358	2.0689	1.4796	1.7035	1.3630
3	2.5370	1.3370	3.2273	1.4763	2.6884	1.3651
4	3.5958	1.3332	4.5951	1.4777	3.8123	1.3618
5	4.9601	1.3351	6.2758	1.4790	5.2476	1.3637

Average: 1.3356 ± 0.0016 Average: 1.4779 ± 0.0014 Average: 1.3637 ± 0.0014

5. Discussion

Now, we examine how the error in the measured values of Δd_m affects the determination of the refractive index. From Eq. (11) it follows that

$$\partial n_2/\partial\Delta d_m = \frac{\left(w^2 m\lambda_0/S\right)\sqrt{\left(1-m^2\lambda_0^2/S^2\right)^3}}{\left(wm\lambda_0/S-\Delta d_m\sqrt{1-m^2\lambda_0^2/S^2}\right)^3\sqrt{1+w^2\left(1-m^2\lambda_0^2/S^2\right)\Big/\left(wm\lambda_0/S-\Delta d_m\sqrt{1-m^2\lambda_0^2/S^2}\right)^2}}. \tag{15}$$

By taking experimental values of the refractive index of Table 1, for the case of the water, we find that the maximum value, obtained from Eq. (15), corresponds to the order m = 1, namely $\partial n_2/\partial\Delta d_1$ = 0.5758 mm^{-1}, and the minimum value corresponds to the order m = 5, which is $\partial n_2/\partial\Delta d_5$ = 0.0970 mm^{-1}. Now, we examine the error in n_2 for the case of the maximum value obtained from Eq. (15), which corresponds to the order m = 1 ($\Delta d_m = \Delta d_1$). For this case, if the error in Δd_1 is 0.001 mm, the error in n_2 is ~0.0006. Then, when Δd_1 = 0.7838 or 0.7858 mm, the value of n_2 is 1.3365 or 1.3377 from Eq. (11). It can be shown, that if the error in the measurement of the vertical separation Δd_m is of one pixel (6.7 μm), the second decimal of the value of the refractive index remains unchanged for all diffraction orders, except for the largest error in the measurement of the first-order.

The error in the angular setting is obtained from Eq. (9):

$$\partial n_2/\partial I = \frac{A_m\left(m\lambda_0\cos I\right)}{B_m^2\left(2S^2P_mQ_m\right)}. \tag{16}$$

where

$$A_m = -2w^2S^3+Q_m\left\{\begin{array}{l}2w\Delta d_m\left[m\lambda_0\left(S^2+2m^2\lambda_0^2-3S^2\cos 2I\right)-S\sin I\left(S^2-6m^2\lambda_0^2+S^2\cos 2I\right)\right]\\ \quad +SP_m\left[S^2\left(2w^2+\left(\Delta d_m\right)^2\right)-2m^2\left(\Delta d_m\right)^2\lambda_0^2+S\left(\Delta d_m\right)^2\left(S\cos 2I-4m\lambda_0\sin I\right)\right]\end{array}\right\},$$

$$B_m = w\left(m\lambda_0+S\sin I\right)-SP_m\left(\Delta d_m+wQ_m\sin I\right),$$

$$P_m = \sqrt{1-\left(m\lambda_0/S+\sin I\right)^2},$$

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and

$$Q_m = \sqrt{1+\left[\Delta d_m/w-(m\lambda_0/S+\sin I)\Big/\sqrt{1-(m\lambda_0/S+\sin I)^2}\right]^2}.$$

For I = 0 (normal incidence), we find that the maximum and minimum values obtained from Eq. (16) are –20.2692 rad^{-1} and –4.6856 rad^{-1}, which correspond to the orders m = 1 and m = 5, respectively. Then, if the error in the angular setting is of 0.1° = 1.745 x 10^{-3} rad (I = 1.745 x 10^{-3} rad), the errors in n_2 are ~0.0354 for m = 1 and ~0.0082 for m = 5. Since this error is large, the angular setting is very important. We find the position for which I = 0 with the following procedure: before placing the cell, the location of the centroid of the spot of the laser on the CCD is recorded. Next, the cell was placed and, whit the laser beam impinging on the diffraction grating, it is rotated until the location of the centroid of the spot of the zeroth-order is located at the position recorded on the CCD before placing the cell.

6. Conclusions

In conclusion, we have presented a very simple method for measuring the refractive index of liquids using a diffraction grating engraved on the inner face of one of the glass walls of a rectangular cell by fs laser ablation. When a laser beam impinges normally on the grating engraved, the diffraction orders produced are deviated when they pass through the cell. The position of the spots of the diffraction orders emerging of the cell is measured in two cases: when the cell is empty and when it is filled with the liquid. By measuring the vertical separation between the spot of m order for the air and the spot of m order for the liquid, we can determine the refractive index of the liquid. The central point of each spot was obtained by the digital image processing of each image, by applying a median filter and finding the mass center. One advantage of this method is that in the measurement of the vertical separation is not necessary to know the thickness and refractive index of the glass walls of the cell. An excellent agreement between the measured and published refractive index values of water, castor oil and ethanol was obtained. The proposed method is very simple, reliable, repeatable, and robust, as required, for example, in many industrial processes.

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