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Five-wavelength laser microrefractometer

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In this paper, the design and testing of a five-wavelength laser microrefractometer are presented. Five semiconductor lasers are used for the spectral region of 405–1320 nm. The presented device is based on the critical angle method. In this case, the critical angle of total internal reflection is determined with the help of a CCD camera detecting the disappearance of the diffraction pattern, created by a metal diffraction grating. The samples of a thin liquid layer (< 10 μm) are placed between a flint-glass prism and a chromium diffraction grating. The refractive indices of two matching liquid products of Cargille Laboratories are investigated for the approbation of the presented device. The measured values of the refractive indices are used for the dispersion curves construction. The obtained values of the refractive indices are compared with the catalog data given by the manufacturer.

Keywords: refractometer, semiconductor lasers, dispersion, refractive index, diffraction.

1. Introduction

The measurement of the refractive index of a small amount of liquid (less than 1 μl), or thin films (with thickness less than 10 μm) is a real experimental challenge. In order to solve this problem, a refractometric method is proposed [1, 2]. The principle of the measurement is based on the critical angle determination by detecting the position where the disappearance of the diffraction pattern takes place. The solid or liquid sample is placed between the reflection (metal) grating and the heavy glass prism. When the incident angle of the laser beam is smaller than the critical angle, determined by the refractive index ratio of the sample and the prism, the beam transmitted through the samples and diffraction pattern is observed. In the case when the incident angle is equal to the critical one, the totally internal reflected beam is observed. The critical angle value is defined as

$$\varphi_c = \sin^{-1}\left(\frac{n}{N}\right) \tag{1}$$

where N and n are the refractive indices of the prism and the sample, respectively.

The method is tested in the wide spectral range – from ultraviolet 266 nm to near-infrared 1064 nm [3]. The basic laser in measurements was a pulled single-mode Nd:YAG laser emitting at the wavelength of 1064 nm. The second, third and fourth harmonics have been used in the experiment. Soon after, an automatic diode laser microrefractometer was reported [4]. Three lasers are mounted on the rotating platform. A further development of this construction is reported in another publication [5].

The main disadvantage of the mentioned above construction [4, 5] is a rotary laser head which needs an additional fine adjustment.

In the present note, the design and the testing of a five-wavelength laser microrefractometer (FWLMR) without moving optical elements are reported.

2. Construction details

The semiconductor lasers used are listed in Table 1.

T a b l e 1. Semiconductor lasers used in FWLMR.

Wavelength [nm]	Manufacturer	Power [mW]
405	Sharp	20
532	ThorLabs	5
656	Sanyo	5
910	ThorLabs	30
1320	Mitsubishi	30

The principle scheme of the FWLMR is shown in Figure 1. The first beam combiner (4) is used to collect the visible laser beams (1 and 2) into one beam (4a). The second beam combiner (5) is used to collect beams from infrared lasers 910 nm and 1320 nm (6 and 7) into one beam (5a). The beam splitter (8) is used to collect all

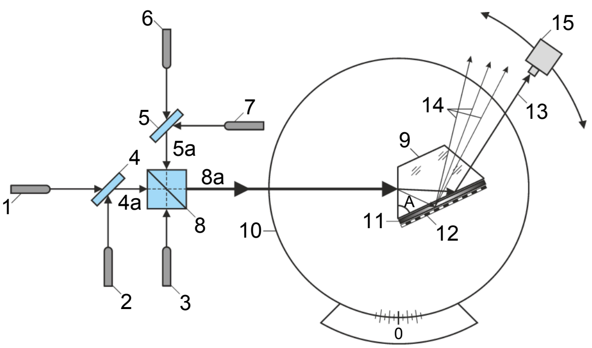

Fig. 1. Principle scheme of the five-wavelength laser microrefractometer: 1 – 405 nm laser, 2 – 656 nm laser, 3 – 532 nm laser, 4 and 5 – beam combiners, 6 – 910 nm laser, 7 – 1320 nm laser, 8 – beam splitter, 9 – glass prism, 10 – rotary stage with vernier, 11 – sample, 12 – metal diffraction grating, 13 – total internal reflected beam (TIR) beam, 14 – diffraction beams, 15 – CCD camera.

five laser beams (4a and 5a) into one beam (8a). It is interesting to note that the green laser (532 nm) (3) is collected by the same beam splitter (8). Despite the inevitable Fresnel losses, the energy is sufficient for a reliable detection of the green diffraction pattern. Two small diaphragms placed at a 50 cm distance from each other, on the 8a beam path, ensure the overlapping of all beams. A heavy-glass prism (9) made of TF-4 with the refracting angle $A = 64.75°$ and a chromium diffraction grating (12) with the period $\Lambda = 20$ μm are used for a critical angle determination, which value is measured by the vernier of the rotary stage "microcontrol" (10) with 1 arcmin resolution. The disappearance of the diffraction pattern is fixed by the CCD camera (15).

3. Results and discussion

The refractive indices of two certified refractive index liquids produced by Cargille Laboratories – non-drying immersion oil for microscopy (cat. no. 16482) and high refractive immersion oil (cat. no. 1812Y), are investigated [6, 7].

The critical angle φ is measured in the air at chosen wavelength and the refractive index of the sample n is calculated by the following formula:

$$n = N\sin\left\{A \pm \arcsin\left[\frac{\sin(\varphi_c)}{N}\right]\right\} \tag{2}$$

where N is the refractive index of the prism for the wavelength used (Table 1).

The main source of experimental uncertainty is the used rotary stage with $\Delta\alpha = 1$ arcmin resolution. Using Equation (1), the experimental uncertainty can be estimated as

$$\Delta n = N\Delta\varphi_c\cos(\varphi_c) \tag{3}$$

In our case, since the obtained values of the critical angle are of the order of 60°, we have $\Delta n = \pm 5\times 10^{-4}$ for the experimental uncertainty.

The optical properties of the materials are usually presented by their dispersion curves. For the most optical materials, far from the fundamental absorption band, the dispersion dependence of their refractive index can be built using the Sellmeier dispersion equation, if they are non-magnetic (*i.e.* $\mu = 1$) [8]. The transmittance of the two investigated liquids was measured by a high precision Perkin Elmer Lambda 19 UV–VIS–NIR spectrophotometer in the spectral range 400–1400 nm to an accuracy of 0.1% and no absorption peaks were detected. The values of the coefficients s and λ_s, the so-called Sellmeier coefficients, can be obtained using the following relation [8]:

$$n^2 - 1 = \frac{s\lambda}{\lambda^2 - \lambda_s^2} \tag{4}$$

In this paper, the values of Sellmeier's coefficients are obtained by the nonlinear model fitting using relation (4) with the set of experimentally determined values of the refractive index with the help of the computational software Wolfram Mathematica. The confidence level is 0.95. Using the coefficients s and λ_s, the dispersion curves of the refractive index are built by the approximated values in the spectral range 400–1400 nm.

The measured values, approximated values and catalog data given by the Cargille Laboratories for the refractive index of two oils are listed in Tables 2 and 3. The uncertainty of the measured refractive index and catalog refractive index is marked as Δn. The difference between the measured refractive index and the catalog refractive index and the approximated refractive index is marked as ΔN.

The results presented in Tables 2, 3, and Figs. 2, 3 show a good agreement between the measured and catalog refractive index values that demonstrate the high precision of the proposed five-wavelength laser microrefractometer.

The main advantages of the proposed device over others are:

1. Rapid measurement;

2. Accuracy – if the resolution of the rotary stage is 1 arcmin, the experimental uncertainty is 5×10^{-4};

3. Opportunity for measurements of turbid and dispersed samples (for example nanoparticles dispersed in water [9], milk and others);

T a b l e 2. Data of non-drying immersion oil for microscopy.

λ [nm]	Measured values $\Delta n = \pm 0.0005$	Catalog data $\Delta n = \pm 0.0002$	Approximated values	ΔN
405	1.5463	–	1.5463	0.0000
532	1.5196	–	1.5198	0.0002
546.1	–	1.5180	1.5192	0.0012
589.3	–	1.5150	1.5148	–0.0002
656	1.5101	–	1.5097	–0.0004
910	1.4991	–	1.4996	0.0005
1320	1.4935	–	1.4940	0.0005

T a b l e 3. Data of high refractive immersion oil.

λ [nm]	Measured values $\Delta n = \pm 0.0005$	Catalog data $\Delta n = \pm 0.0002$	Approximated values	ΔN
405	1.7670	–	1.7675	–0.0005
486.1	–	1.7273	1.7268	0.0005
532	1.7127	–	1.7126	0.0001
589.3	–	1.7000	1.6999	0.0001
656	1.6900	1.6900	1.6895	0.0005
910	1.6694	–	1.6695	–0.0001
1320	1.6589	–	1.6587	–0.0002

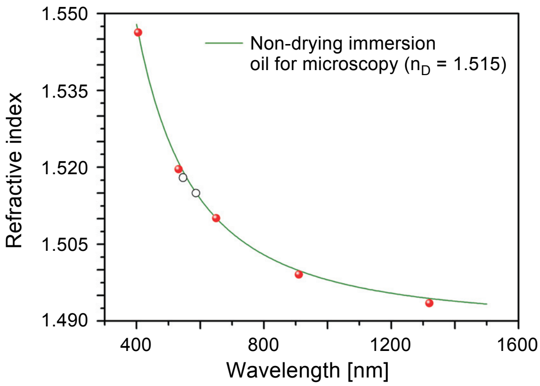

Fig. 2. The dispersion dependence for non-drying immersion oil for microscopy. The unfilled symbols are catalog data given by the Cargille Laboratory.

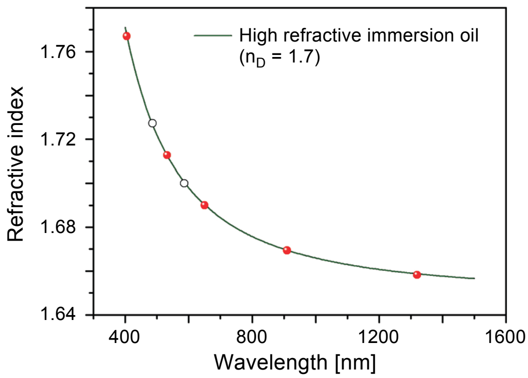

Fig. 3. The dispersion dependence of the liquid with high refractive index. The unfilled symbols are catalog data given by the Cargille Laboratory.

4. Possibility of refractive index measurement of liquid samples with small volume – under 4 µl.

5. Possibility of refractive index measurement of solid films and layers with thickness under 100 µm;

6. Refractive index measurement in the wide spectral range – near UV, VIS and near IR;

7. Small dimensions.

4. Conclusion

The present paper describes the design of a five-wavelength laser microrefractometer as a laboratory device for determination of the refractive indices of optical materials (liquids and solid films) in a wide spectral range including the whole visible and near infrared region. The measurement is improved with the CCD camera for determination of the critical angle at different laser wavelengths. The experimental uncertainty is less than 5×10^{-4} and it depends generally on the rotary stage used.

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