Compact liquid-refractive index measuring equipment

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Based on total reflection principle, a compact liquid-refractive index measuring equipment was designed and fabricated, in which a diode laser was used as light source and a charge-coupled device (CCD) as photodetector. The influence on measurement accuracy of the wavelength shift and intensity fluctuation of the diode laser were surmounted by an effective feedback method. It is crucial whether the diode laser could be used in such a system.

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Change-coupled device (CCD) plays very important role in making a conventional optic instrument miniaturized and brain power passessed. A compact liquid-refractive index measuring equipment is very useful for real-time monitoring in industrial production. Some researches have been done in this field, in which CCD was used as the photodetector[1,2]. The key problem for such equipment is that the intensity fluctuation of the light source will bring large measuring error. Adopting diode laser as a light source, we took effective means to well solve this problem. The measuring accuracy was also well improved.

The schematic of measuring principle is shown in Fig. 1.

The light source sends out a light beam with a proper range of incident angles and falls on the interface between the measuring prism and the liquid to be measured. The incident light with angles smaller than critical angle will partially reflect and partially refract, while the incident light with angles larger than the critical angle will total reflect. It will form a dark region and a bright region with a boundary line between them on CCD, see Fig. 1. The dark region corresponds to the incident angles is smaller than critical angle and the bright region corresponds to the incident angles is larger than the critical angle. When the refractive index of the liquid changes, the critical angle and the boundary line also change correspondingly. The refractive index is measured from the position of the boundary line. The measuring principle is just like that used in Abbe refractmeter. Such liquid which can be measured by Abbe refractometer can also be measured by this equipment. The difference is that the human's eye is replaced by CCD.

We utilized a 5-mW diode laser (λ = 670 nm) as the light source. Diode laser has long life, good monochromaticity, low operation voltage and small spatial volume. It is the utilization of diode laser that makes the measuring equipment compact. The big problem of using diode laser is that the light intensity will fluctuate largely when temperature varies. It will directly affect the measurement accuracy. How can the light intensity fluctuation and shift affect the measuring accuracy? As we know the boundary line between dark and bright regions is not step, but gradual (see curve *db* in Fig. 2). If the light intensity is not stable, the boundary line can not be determined accurately, as we can see in Fig. 2.

In Fig. 2, abscissa N is the number of the photosensitive elements on CCD, and ordinate m is the gray levels of CCD which is 255.

Figure 2 came from an actual measurement. In Fig. 2, curve *abc* is the light intensity distribution on CCD when liquid to be measured did not exist. Curve *dbc* is the light intensity distribution during measurement. Point A corresponds to the boundary line, and B is the point at which the sensitive element is just saturanted.

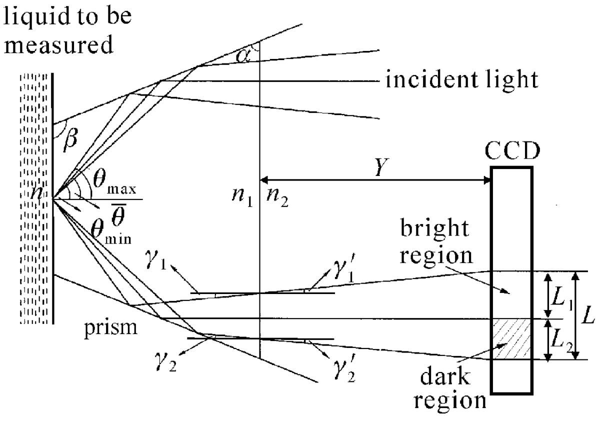

Fig. 1. The schematic of measuring principle.

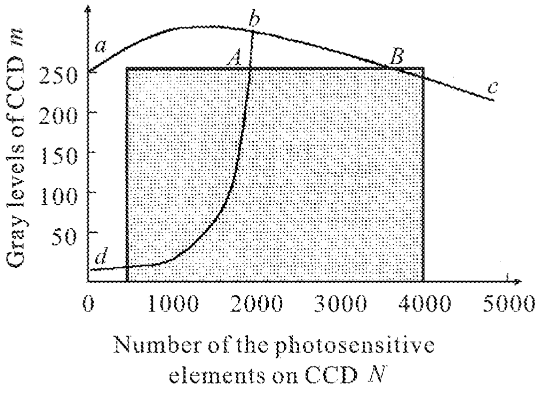

Fig. 2. The light intensity distribution on CCD.

We can see that if the light intensity changes, curve *abc* will go up or down, and then points A and B will move towards right or left and so the measured data which is determined by the position of A changes also. It means that, if the light intensity changes, although for the same refractive index, the measured data will vary. In our method, we took the sample light from the photosensitive element at point B and stabilized the light intensity by using negative feedback process and then made B fixed. It is equivalent to that the intensity level has a reference, and the intensity should be stabilized around this reference. It is an effective method to offset the intensity fluctuation, particularly for long-time stability. Now the question is to what degree should the intensity fluctuation be reduced in order to ensure the measuring accuracy of order 1×10^{-4}? From Fig. 2, we can see that $\angle bBA$ is about 20°, and $\angle bAB$ is 80°. When the curve *abc* goes up or down due to the light intensity fluctuation, the ratio of the displacements towards right or left of the points B and A is $\cot \angle bAB / \cot \angle bAB \approx 15.6$. It means that if the point B moves towards right (or left) from its original position no larger than 7 elements, the point A can keep in its original position. In such case, the measurement is not affected by the light intensity fluctuation.

In the experiment we took the 4500th element as the reference, which can be done by adjusting the light intensity. The point B was restricted in the range between 4496th and 4504th elements. If the point B was not in this range, the microprocessor will send pulses to digital potentiometers to change their resistance, which determined the injection current of the diode laser. Two similar digital potentiometers were used in series, each with 32 tapping points. The first one was used to adjust the injection current coarsely, and the second for fine adjustment. That is to say, the intensity fluctuation could be reduced to 1/1024.

In the experiment, the measuring prism is made of K9-glass with $n_{670\,\mathrm{nm}} = 1.51348$ for wavelength of 670 nm. Angle α of the prism (see Fig. 1) is determined by the measurement range of the refractive index. Let $\theta_{\min}$, $\bar{\theta}$ and $\theta_{\max}$ are the critical angles corresponding to the minimum, middle and maximum values of the refractive index $n_{\min}$, $\bar{n}$ and $n_{\max}$, then

$$\bar{\theta} = \theta_{\min} + \frac{\theta_{\max} - \theta_{\min}}{2}. \qquad (1)$$

The incident light with angle $\bar{\theta}$ will emerge vertically to the surface of the prism. From Fig. 1 and Eq. (1),

$$\alpha = \frac{180^\circ - \bar{\theta}}{2}. \qquad (2)$$

Now we calculate the distance Y from the surface of the prism to CCD (see Fig. 1), where $\gamma_1 = \bar{\theta} - \theta_{\min}$, $\gamma_2 = \theta_{\max} - \bar{\theta}$, γ_1' and γ_2' are the refractive angles in air. From Fig. 1,

$$Y = \frac{L}{\tan \gamma_1' - \tan \gamma_2'}. \qquad (3)$$

CCD used in the experiment has 5430 basic elements, one effective sensitive element is 7 μm, and the total size of CCD is 37.38 mm. From Fig. 1 we can see the relation between the number of the elements and the refractive index n which is to be measured.

When $n_{\min} < n < \bar{n}$,

$$N = N_0 + \frac{1}{7 \times 10^{-6}} \times \left\{ L_1 - Y \tan[\arcsin(\frac{n_1}{n_2} \sin(\bar{\theta} - \arcsin \frac{n}{n_1}))] \right\}, \qquad (4)$$

when $\bar{n} < n < n_{\max}$,

$$N = N_0 + \frac{1}{7 \times 10^{-6}} \times \left\{ L_1 + Y \tan[\arcsin(\frac{n_1}{n_2} \sin(\arcsin \frac{n}{n_1} - \bar{\theta}))] \right\}, \qquad (5)$$

where N_0 is the number of the elements when $n = n_{\min}$, $n_2 = 1.0003$ is the refractive index of air, $n_1 = 1.51348$. From Eqs. (4) and (5), we can see that to the different value of n, there is a correspondent value of N, so we can calculate refractive index from measured N through Eqs. (4) and (5). The refractive index is also the function of temperature, so we must measure the temperature simultaneously and make an appropriate compensation. We should also take into consideration about wavelength shift of a diode laser due to the temperature variation, typically 0.3 nm/°C. The value of n_1 is equal to 1.51348 used in Eqs. (4) and (5) with temperature of 20 °C. When temperature changes, we should correct n_1 correspondingly.

In the experiment, the measuring rang of n is 1.3330−1.3820. The shape of the equipment is a cylinder, $\Phi 125 \times 255$ mm, see Fig. 3. When the refractive index of the liquid to be measured changed for $\Delta n = 1 \times 10^{-4}$, correspondingly, the boundary line on CCD moved across 8 elements on an average.

Thus the resolution of this system is $\Delta n = 1.25 \times 10^{-5}$. Because of the machining precision and setting accuracy the measuring accuracy is 1×10^{-4} in practice. Without the negative feedback procedure, the intensity fluctuation (including slow shift mainly due to the temperature variation) was about 8%. Correspondingly, the measuring accuracy was only 1×10^{-2}.

When a LED or a tungsten halogen was used as the light source, except the intensity fluctuation, the bandwidth also affected the measurement, and the measuring accuracy was 0.1%[1,2].

The standard sample here used was provided by

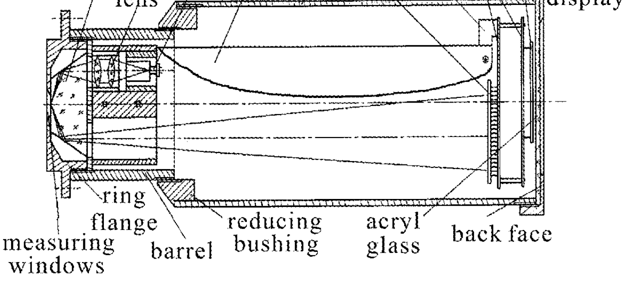

Fig. 3. The measuring equipment.

Table 1. Comparison Between the Data from Experiments and the Data of Standard Sample

Data from Experiments	Data of Standard Sample
1.3329	1.3330
1.3374	1.3374
1.3455	1.3456
1.3581	1.3582
1.3681	1.3682
1.3767	1.3765
1.3810	1.3811

Heilongjiang Metering Institute. The experimental data in Table 1 were the average values, but among all the data from the experiments, the maximum errors were not larger than $\pm 1 \times 10^{-4}$. We have done the measurements every day for six months, the temperatures were from 10 to 35 °C, the working state of the equipment is very stable.

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References

1. C. Tan, S. X. Zhu, J. P. Zhong, Y. Xiao, and W. Zeng, Sugarcane Sugar Manufacture (in Chinese) (2) 34 (1987).
2. Z. Xiao, G. C. Liu, Z. Y. Lei, Y. S. Lu, W. H. Chen, E. Y. Guo, and H. Xue, Microelectronic and Computer (in Chinese) **14** (3), 49 (1997).