16.6. Interference microrefractometry of liquids

If the refractive index n and the thickness t of a solid plate-like object (Fig. 16.71) are known, the refractive index n' of a liquid that surrounds the object can be determined from the formula $\delta = (n' - n)t$ by measuring the optical path difference δ. However, for microinterferometry of liquids special glass plates or microchambers are available from the manufacturers of microinterferometers.

16.6.1. Determining the refractive index of liquids from optical path difference measurements

Different microchambers may be used for determining the refractive index of liquids from the formula mentioned above. The most popular is that available commercially from VEB Carl Zeiss (Jena) as the standard equipment of the interference microscopes from the interphako family (Peraval-interphako, Jenaval-interphako, Jenavert-interphako, and others). This microchamber is simply a glass plate with an arched groove (Fig. 16.74a) of trapezial cross-section (Fig. 16.74b). If an interphako microscope or the Biolar PI microinterferometer is used, then two completely sheared images of the groove can be obtained using a low-power objective (10 ×). In these images the interference fringes are displaced symmetrically in the opposite directions (Fig.

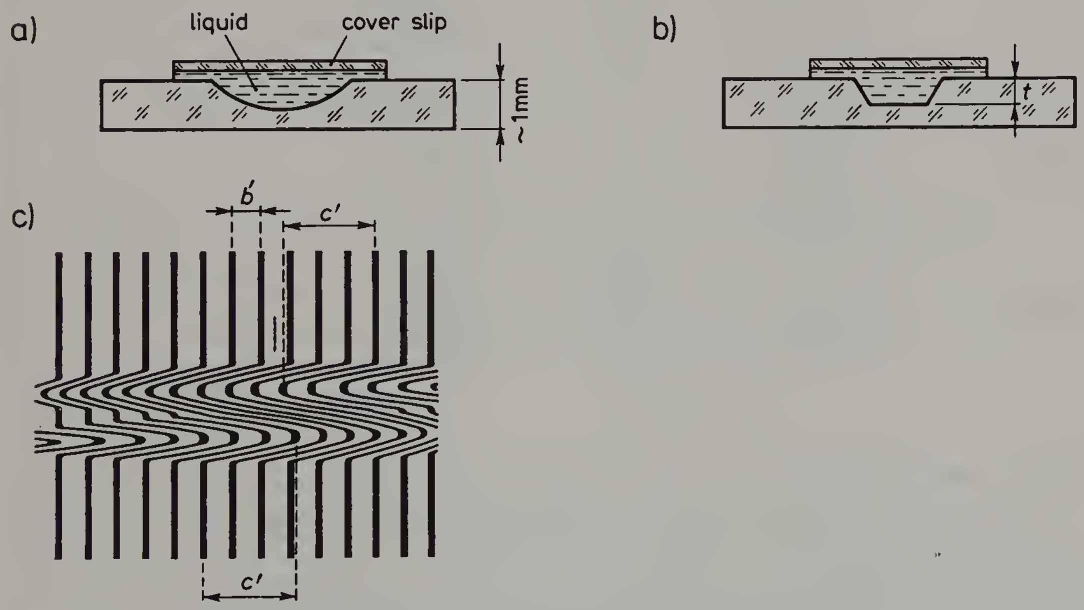

Fig. 16.74. Wavefront shear interference microrefractometry by using the Zeiss (Jena) microchamber.

16.74c). To determine the refractive index n' of a liquid, the interfringe spacing b' and the fringe displacement c' or $2c'$ are only measured. The index n' follows from the relation $\delta = (n'-n)t = c'\lambda/b'$, where λ is the wavelength of light used and t is the depth of the groove at its central point marked by a reference line. The trapezial cross-section of the groove enables the continuous connections to be easily observed between the displaced and undisplaced interference fringes, thus no problems in identifying the interference orders arise.

Another more accurate procedure is based on the measurement of the interfringe spacing b and the fringe displacement c (or $2c$) by means of a phase compensator or by using the phase screw (*PS*, Fig. 16.45) if the Biolar PI microinterferometer is employed (see Eq. (16.27) and the related text).

Not only the groove depth but also the spectral dispersion of the refractive index n of the microrefractometric plate must be known. The respective data for performing a plot $n(\lambda)$ are delivered by the manufacturer (see Table 16.12).

TABLE 16.12
Refractive indices (n) pertaining to the wavelengths of the Fraunhofer spectral lines F, e, d, and C, respective ratios $\Lambda = \lambda/(n-1)$, and the groove depth (d) of a standard set of refractometric plates from C. Zeiss Jena

Plate (Factory number)	Wavelength λ [nm]	n	Λ [nm]	d [μm]
I (620575)	486.1 (F)	1.51703	940.17755	10.0230
	546.1 (e)	1.51333	1063.83808	
	587.6 (d)	1.51133	1149.16003	
	656.3 (C)	1.50893	1289.56831	
II (620561)	486.1	1.51989	935.00548	12.5425
	546.1	1.51619	1057.94378	
	587.6	1.51419	1142.76824	
	656.3	1.51179	1282.36191	
III (620566)	486.1	1.52470	926.43415	13.3762
	546.1	1.52100	1048.17658	
	587.6	1.51900	1132.17726	
	656.3	1.51660	1270.42199	
		$\Delta n = 0.0002$		$\Delta d = 0.005d$

The plot enables the refractive index n to be readily read out for a given light wavelength λ used in the measuring process. Frequently it is more convenient to use a plot $\Lambda(\lambda)$, where $\Lambda = \lambda/(n-1)$. The latter is almost linear (Fig. 16.75) and thus very suitable for processing using a PC computer.

To determine the refractive index n' with an accuracy $\Delta n' = \pm 10^{-4}$ using the Zeiss microrefractometric plates (Table 16.12), the optical path difference

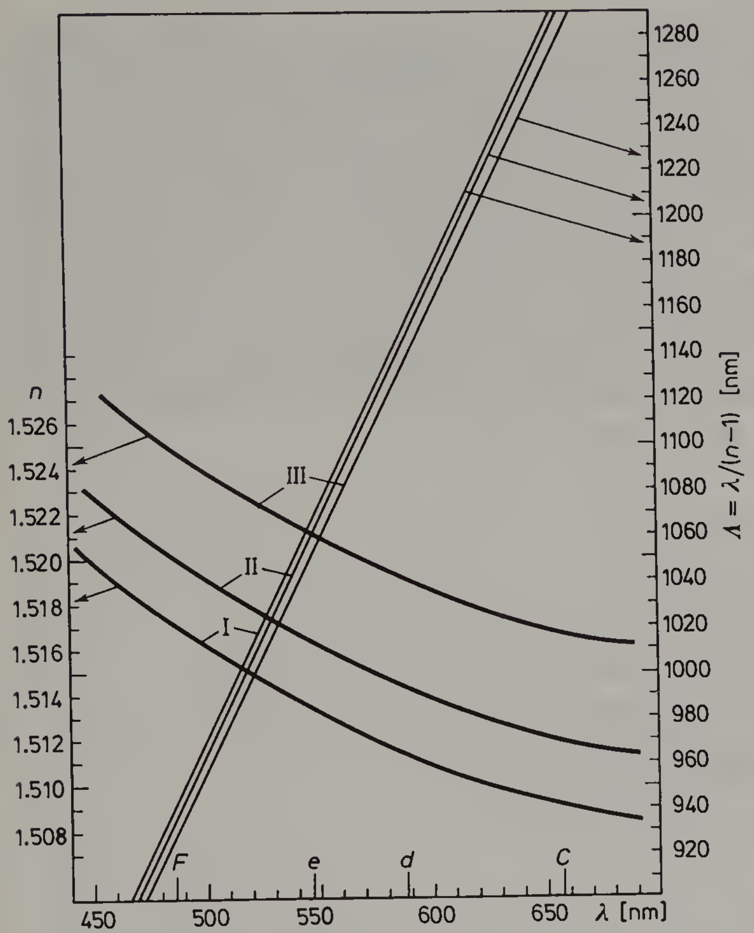

Fig. 16.75. Refractive index dispersion $n(\lambda)$ and $\Lambda(\lambda)$ of the Zeiss (Jena) refractometric microchambers (see also Table 16.12).

δ must be measured with an accuracy $\Delta\delta = \pm\lambda/500$. Such a measuring accuracy is not possible when the conventional fringe-field techniques are used (see Subsection 16.3.1), but such an accuracy level can be achieved if the VAWI-1 technique is employed (see Subsection 16.4.1). Moreover, this technique permits us to determine not only the refractive index n' for a given light wavelength but also the spectral dispersion $n'(\lambda)$ across the visible spectrum (see Ref. [16.111]).

16.6.2. Determining the refractive index of liquids from measurements of the optical path difference gradient

An optical path difference gradient is produced by a refractometric plate *RP* with a prismatic end which, together with an auxiliary plate *AP*, constitutes a semi-trapezial channel for the examined liquid between a microscope slide

MS and a cover slip *CS* (Figs. 16.76a and b). Such a microchamber belongs to the additional equipment of the Biolar PI microinterferometer (Fig. 16.45). Normally, the objective PI 10 × is used, whose birefringent prism W_0 is crossed with the tube birefringent prism W_2 No.2, and the microchamber is placed so that its top edge *E* is in focus and oriented at right angles to interference fringes observed in the image plane Π'. The duplicated images E'_1 and E'_2 of the edge *E* divide the field of view into two halves (Fig. 16.76c). One half contains the undisplaced fringes *I* and the other is occupied by fringes *I'* displaced by the prismatic part of the refractometric plate *RP*. Both fringe families are connected by intermediate interference fringes which cross the central part of the field of view between the edge images E'_1 and E'_2. Thus no problem arises in identyfying the orders of the displaced fringes *I'*.

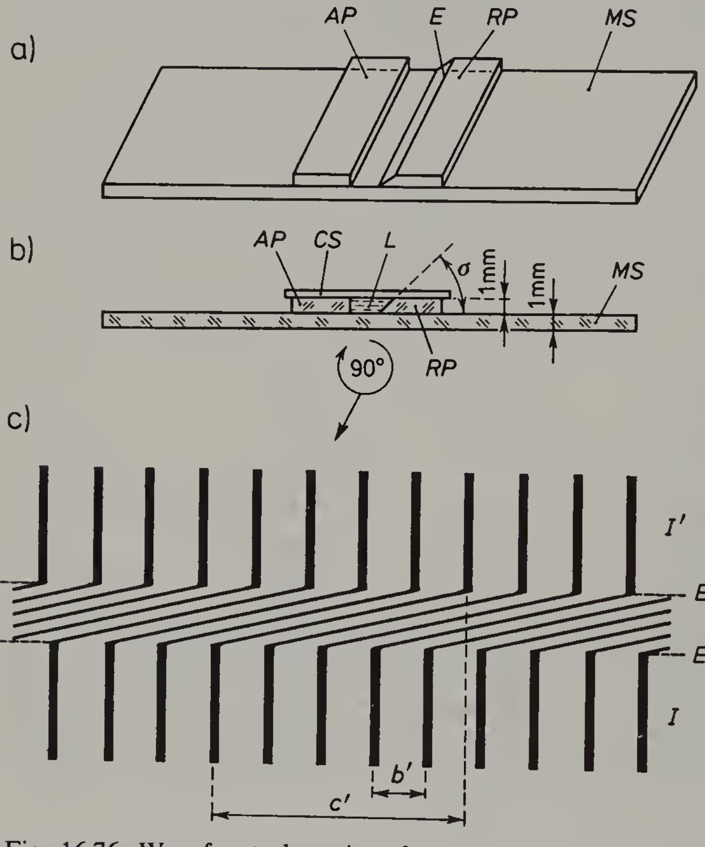

Fig. 16.76. Wavefront shear interference microrefractometry by using a microchamber with a prismatic glass plate.

The refractive index n' of the liquid *L* is determined from the following formula:

$$n' = n \pm \frac{\Delta}{s \tan\sigma \sin\zeta} \tag{16.129a}$$

where Δ is the optical path difference produced by the prismatic part of the refractometric plate RP betweeen the interfering waves of monochromatic light, s is the resultant wavefront shear (see Fig. 16.41b or d), σ is the prism angle (Fig. 16.76b), ζ is the angle between the edge E of the plate RP and the direction of the wavefront shear s, and n is the refractive index of the plate RP. From the geometry of Fig. 16.41b or d it follows that $s \sin\zeta = s_0$. The angle σ is simply equal to 45° and the optical path difference may be expressed as $\Delta = c\lambda/b$, where b and c are the interfringe spacing and the fringe displacement, both measured by means of the phase screw PS (Fig. 16.45). The ratio c/b is of course equal to c'/b' (Fig. 16.76c). Consequently, Eq. (16.129a) can be rewritten as

$$n' = n \pm Ac, \tag{16.129b}$$

where A is the constant term for a given wavelength of monochromatic light ($A = \lambda/bs_0 \tan\sigma$).

It is worth noting that Eqs. (16.129) follow directly from Eq. (7.18) dealing with DIC microinterferometry (see Subsection 7.1.3 in Volume 2). As can be seen, the relationship between n' and c is linear and the measuring technique in question is suitable for an accurate calibration using only a single standard liquid whose refractive index is precisely known. A stright-line plot $n'(c)$ is determined by two points, one of which is given by $n' = n$ and $c = 0$, and the other by $n' = n'_M$ and $c = c_M$, where n'_M and c_M are the refractive index and the fringe displacement for a master calibration liquid (available, e.g., from Cargille Laboratories).

The constant parameter A in Eq. (16.129b) is slightly dependent on light wavelength; thus the function $A(\lambda)$ and, of course, the spectral dispersion $n(\lambda)$ of the refractive index of the refractometric plate RP (Fig. 16.76) must be known if the spectral dispersion $n'(\lambda)$ of the refractive index of a liquid under study will be measured.

It is self-evident that the sign "+" is taken for $n' > n$ and the sign "−" for $n' < n$ in Eqs. (16.129).

It is also important to note that the microchamber shown in Fig. 16.76 is also very convenient for the study of polymers, cements, and other adhesive substances [16.135, 16.136] (see also Ref. [1.31] cited in Volume 1).

A gradient microchamber for interference microrefractometry of liquids was also constructed by S. Iversen and F. H. Smith [16.137].[31] That consists of two semicircular glass plates whose diametrical surfaces are optically worked and maintained in close mutual contact to form a disc. A quadrant of one of

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[31] This reference is due to courtesy of J. Sikorski who used frequently the Iversen and Smith microchamber in his research work at Textile Physics Laboratory, The University of Leeds, England (personal communication).

the semicircular plates is optically worked to form a 10° wedge. As the thickness of the liquid layer is continuously increasing along the wedge, a system of evenly spaced fringes will be observed when the microchamber is viewed through a conventional interference microscope having a reference wavefront, or also through a double-refracting interference microscope with large wavefront shear. The spacing of the fringes is a function of the refractive index n' of the liquid. By determining the interfringe spacing, the index n' is readily obtained from a linear calibration plot. An accuracy of at least $\Delta n' = 0.001$ was reported [16.137].

The above-mentioned fringes can be compared to those shown in Fig. 16.76c as the oblique fringes between E'_1 and E'_2, which connect the displaced (I') and undisplaced (I) fringes. The interfringe spacing of the oblique fringes is, of course, a function of n' and by counting the number (N) of those fringes per unit length along the direction perpendicular to the fringes we can also determine n' in a way similar to that described by Iversen and Smith [16.137]. It is self-evident that a close relationship occurs also between N and the fringe displacement c' which is a functon of n'. On the other hand, the interfringe spacing b' of the undisplaced (I) and displaced (I') fringes is the same, as shown in Fig. 16.76c; no relationship occurs between b' and n'. This is an essential difference between the writer's technique and that described by Iversen and Smith.

16.7. Microinterferometry of thin films

Thin films, layers, microtom sections, and other like objects, are especially suitable for microinterferometric determinations of their thickness and/or refractive indices. Many techniques have been developed for microinterferometry of these objects. Among them, the Tolansky technique based on multiple-beam interference is very popular in practice (see Subsection 16.1.2 and Refs. [16.7], [16.12], [16.14], and [16.15]). However, this technique cannot be effectively used for accurate measurements of transparent films deposited on also transparent substrates without their metallization and thus destruction. On the other hand, two-beam microinterferometry is free from this defect, but its conventional techniques are not sufficiently accurate for quantitative studies of thin films largely used in industrial optics, optoelectronics or semiconductor industry. Some unconventional techniques of two-beam microinterferometry based on the VAWI method (see Section 16.4) will therefore be proposed below for the measurement of the thickness and/or the refractive index of thin films, layers, microtom sections, and other like objects. Frequently these techniques lead to a measuring accuracy comparable with that offered by multiple-beam interferometry and are free from troubles