


A simple method for the determination of refractive indices of (rough) transparent solids

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Simple methods for the determination of refractive indices of transparent polymers and inorganic and organic solids of irregular geometry or with scratched or corrugated surfaces are rare. A classical procedure is based on the invisibility of a body immersed in a liquid with the same refractive index as that of the body. In order to avoid the laborious procedure connected with the search for a liquid with matching refractive index and to find an approach which is independent of the observation by eye, we describe here a modified immersion method which allows the ready determination of the refractive index of solids. The present method is based on the interpolation of the maximum transmission ($n_{T_{\max}}$) of a solid immersed in liquids with different, typically non-matching, refractive indices. Illustrations with quartz glass, crown glass and poly(vinylidene fluoride) (PVDF) films showed that $n_{T_{\max}}$ can be determined with a reproducibility of ± 0.003 . By comparison with refractive indices determined by ellipsometry, it was concluded that the refractive index of a solid can be determined with the modified immersion method within an accuracy better than ± 0.01 when systematic errors resulting from the fit method are also taken into consideration. © 2005 Springer Science + Business Media, Inc.

1. Introduction

The refractive index is a key characteristic of materials used in optics, and, accordingly, a number of methods have been developed in order to measure this quantity [e.g., 1]. In the case of solids, most of these methods are highly demanding with respect to the planarity of the samples on both the microscopic and macroscopic scale. Refractometry with standard equipment is of limited precision for the investigation of solids. Special equipment can enhance the accuracy of refractive indices to the region of ± 0.001 , but is suited only for appropriate sample geometries [2]. Ellipsometry [3] is a precise method for the determination of refractive indices, provided the samples are highly planar and void of scratches. Suppliers of such instruments claim the precision of measured refractive indices to be ± 0.005 [4]. Prism coupling measurements [5, 6] also require high quality sample surfaces, and the accuracy of refractive indices measured is indicated by respective suppliers to be ± 0.001 [7, 8].

Methods which allow the determination of refractive indices of non-planar solids, e.g., solids with rough or corrugated surfaces or samples with irregular geometry, rely on the dependency on the refractive index of the surrounding medium of the extent of light transmitted through the solid. For example, a wavelength-dependent minimum in transmission arises as a consequence of a maximum in Mie scattering, which has

been used for the extrapolation of the refractive index of powders of uniform size [9]. The refractive index of a solid also can be estimated visually when a liquid of close refractive index (n) is found which renders the immersed solid invisible [10–12]. This is illustrated in Fig. 1, where a roughened glass slide ($n = 1.519$) is invisible in silicone oil as a refractive-index-matching liquid ($n = 1.515$), but is visible when in contact with media of a substantially different refractive index, here water ($n = 1.333$) and air ($n = 1.000$). The search for a liquid with a refractive index that matches that of the solid which is to be characterized can, however, be quite laborious. The precision of the immersion method, which under ordinary conditions is limited since the sample may visually disappear already when the refractive indices of solid and liquid still differ, can be improved by use of a phase contrast microscope [13]. In the following, we will describe a convenient, modified immersion method which allows the determination of the refractive index of solids by simple transmission measurements using an ordinary UV/vis spectrometer. The principal advantage of this method is that the search for a refractive index matching liquid is avoided. We note that the immersion method previously has been combined with UV/vis spectroscopy for the determination of the dispersion curve of powders with the help of the dependency of the refractive index on the temperature [14] which is, however, not the subject of this study.

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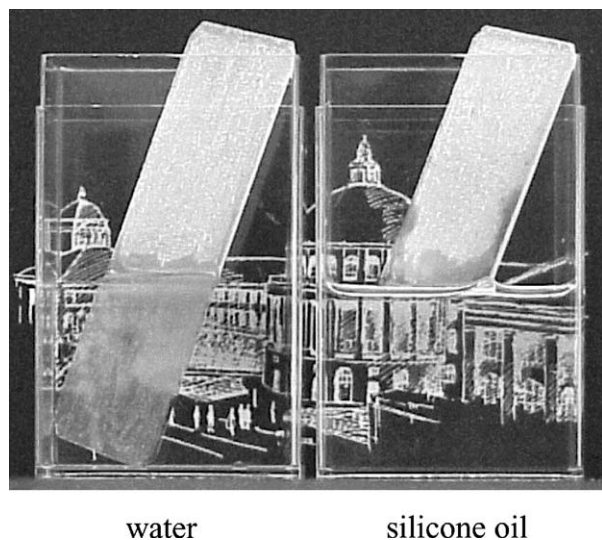


Figure 1 Roughened crown glass slide ($n = 1.519$) immersed in water ($n = 1.333$) and silicone oil ($n = 1.515$).

2. Experimental

Immersion liquids were obtained from Riedel de Haën (Buchs, Switzerland, 4 and 9, Table I) and Fluka (Buchs, Switzerland, all others), and used as received. Quartz glass of 1 mm thickness (Suprasil[®] 1) was obtained from Wisag AG, Zürich (Switzerland), and crown glass of 1 mm thickness (Superfrost[®] microscope slides) from Erie Scientific Company, Portsmouth (New Hampshire, USA). Poly(vinylidene fluoride) (PVDF) films of ca. 50 μm thickness were prepared by compression molding PVDF powder (weight average molecular weight ca. 140 kg/mol, obtained from Polysciences, Warrington PA, USA) at 210°C, followed by quenching to room temperature.

The refractive indices of the various liquids used were determined with an Abbé refractometer (3T, Atago, Tokyo) at 20°C. The liquids had been previously kept at this temperature ($20 \pm 0.1^\circ\text{C}$) for at least 2 h using a Julabo MD 13 thermostat.

UV/vis transmission measurements were performed in quartz glass cuvettes of 1 cm path lengths in the wavelength range of 570–600 nm with a Lambda 900

TABLE I Liquids used as immersion media, and their refractive indices (n) at 589 nm (Na β line) at 20°C, obtained from different sources

No.	Liquid	n supplier	n literature	n this work
1	Water (HPLC quality)	1.333	1.33283 [17]	1.3333
2	Ethanol	1.363	1.3611 [17]	1.3625
2	Isooctane	1.391	1.39145 [18]	1.3919
4	Methylcyclohexane	1.423	1.42312 [18]	1.4234
5	Hexadecane	1.435	1.43455 [18]	1.4350
6	Cyclohexanone	1.450	1.4507 [17]	1.4505
7	<i>Trans</i> -decahydronaphthalene	1.469	1.46932 [18]	1.4697
8	Decahydronaphthalene <i>cis/trans</i> mixture	1.474	1.4752 [17]	1.4743
9	Toluene	1.497	1.49693 [18]	1.4972
10	1,2-dichlorobenzene	1.552	1.5515 [17]	1.5518
11	Dibenzylether	1.562	1.5615 [19]	1.5626
12	1,2,4-trichlorobenzene	1.571	1.5717 [17]	1.5719
13	1-methylnaphthalene	1.614	1.61755 [18]	1.6165

spectrometer (Perkin Elmer) at a rate of 250 nm/min and a slit width of 1 nm. The solid, sheet-like samples were clamped in a self-constructed sample holder, shown in Fig. 2, enabling the installation of a fixed sample geometry in the quartz glass cuvette, which had been filled previously with the respective liquids. The samples were oriented perpendicular to the incident light beam. Spot tests carried out with placing the samples directly in the cuvette (i.e., without the sample holder) did not yield significantly different results. The liquids and solids to be tested were placed in separate test tubes and those test tubes were kept for at least 2 h in a water bath at a temperature of $20 \pm 0.1^\circ\text{C}$. The empty cuvettes were also immersed in a test tube filled with ethanol at a level below the cuvette height and also kept at 20°C for at least 2 h in order to adjust the temperature of the glass walls of the cuvettes. Then, the cuvettes were filled with the liquid selected and again stored for 15 min at 20°C, after which the UV/vis spectrum of the liquid was recorded with respect to air and used as the background spectrum. The solids were then immersed in the same cuvette and kept at 20°C for 5 min; the transmission spectrum was measured again towards air and the background spectrum was subtracted.

Refractive index measurements by ellipsometry were performed by Thomas Wagner at L.O.T.-Oriol GmbH & Co. KG, Darmstadt (Germany), using a spectroscopic ellipsometer of the type VASE[®] from J. A. Woollam Co (Lincoln, Nebraska, USA). The refractive indices were determined at a wavelength of 589 nm using planar specimen.

In order to increase the surface roughness of the glass samples (for subsequent UV/vis transmission measurements), the substrates were abraded on both sides with a rotating corundum abrasive paper mounted on a multi tool model 395 from Dremel (Zuchwil, Switzerland), or with a rotating aluminum oxide grindstone No. 500, mounted on the same tool. The latter was used for the preparation of surfaces with higher roughnesses. Poly(vinylidene fluoride) (PVDF) films were abraded on both sides with corundum abrasive paper of granulation 400. Surface roughnesses were determined with a KLA-Tencor P-11 profilometer. The samples were scanned at a load of 10 mg, a rate of 100 $\mu\text{m/s}$ and a frequency of 200 Hz. The profiles were recorded along the middle on each side of the samples over a distance of 8 mm parallel to the long and 1.5 mm parallel to the short edge. The surface roughnesses R_a and R_q were calculated automatically by the software of the instrument, where

$$R_a = \frac{1}{L} \int_0^L |z| dx \quad (1)$$

and

$$R_q = \sqrt{\frac{1}{L} \int_0^L z^2 dx} \quad (2)$$

with L denoting the scan length, x the position on the scanning axis, and z the vertical distance of the sample surface at the position x related to the average surface

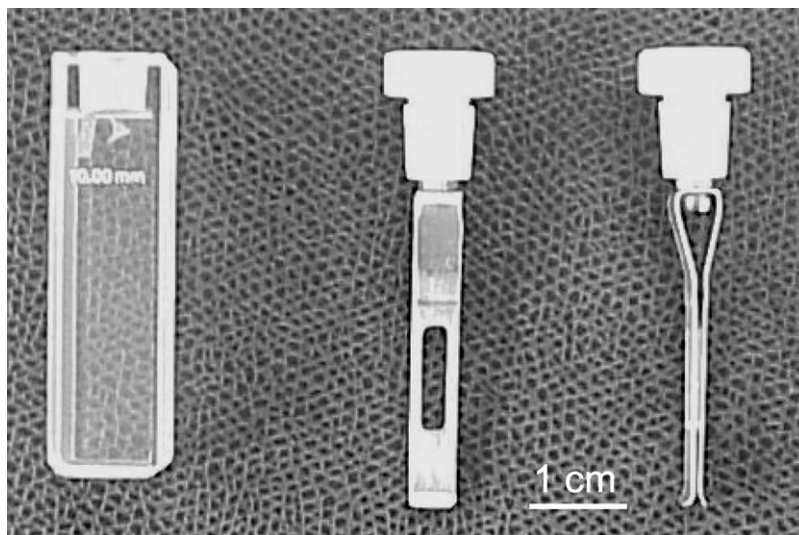


Figure 2 Sample holders used for a defined placement of sheet-like samples in a quartz glass cuvette filled with an immersion liquid.

level of the corresponding sample; by definition

$$\int_0^L z dx = 0 \quad (3)$$

3. Results and discussion

3.1. Model calculations

The refractive index calculations described in the following are based on the dependence of the transmission of light through a solid on the refractive index of the surrounding medium. The transmission becomes maximal when the refractive indices of immersed body and immersed liquid are equal. In the simple case of a plane, non-absorbing dielectric sheet immersed in a non-absorbing dielectric liquid, the transmission T at light incident perpendicular to the solid's surface is given by the equation [15]

$$T = \frac{4n_s n_m}{(n_s + n_m)^2} \quad (4)$$

where n_s and n_m are the refractive indices of the solid and the surrounding medium, respectively. Fig. 3 shows

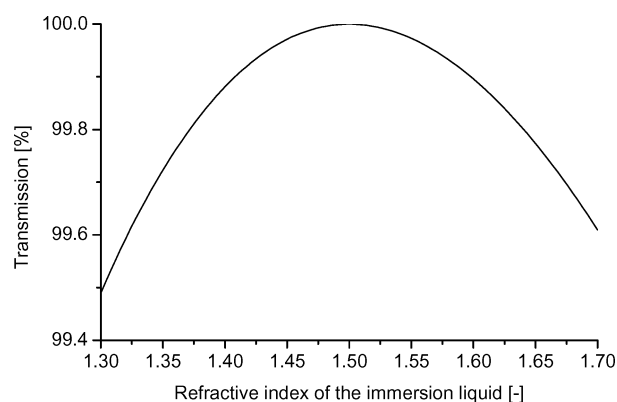


Figure 3 Transmission of a planar sample with $n = 1.5$ immersed in liquids of various refractive indices, calculated with Equation 4, where the sample is oriented perpendicular to the incident light beam.

the results of calculations according to this equation for $n_s = 1.5$ (a typical value of the refractive index region of polymers and many glasses) and $n_m = 1.3$ – 1.7 . Naturally, the transmission maximum is found at $n_m = n_s = 1.5$, and would appear likely that this maximum transmission can be readily interpolated when the transmission is measured with only a few species of different n_m . However, the differences in transmission resulting upon immersion, e.g., at most 0.5% in the above example, are often too small to be detected accurately by a standard UV/vis spectrometer. The difference in transmission could increase markedly if light would impinge onto the sample under a very low angle with respect to the surface, but then the transmission becomes very sensitive to the precise position of the sample.

Differences in transmission can be enhanced at perpendicular light incidence by roughening of the surfaces, since rough surfaces at least partially deflect incident light and, therewith, reduce the amount of light arriving at the detector of a UV/vis spectrometer. Different mechanisms may be involved in light scattering by rough surfaces, as they usually contain high differences on a variety of length scales. However, in order to illustrate the effect of surface roughness on the transmission, a facet model for light transmission through dielectric surfaces [16] is convenient. In this model (see Fig. 4), it is assumed that light is scattered by a rough dielectric interface, characterized by a rms (R_q) roughness, σ , and a surface correlation length, L , that are both larger than the wavelength of light. The local slope s is defined as the angle between the surface normal \mathbf{n} and the average normal \mathbf{z} . Assuming a Gaussian distribution of heights, the local slope s is distributed as:

$$P_s(s) = \frac{L/\sigma}{2\sqrt{\pi} \cos^2(s)} \exp \left[-\frac{(L \tan s)^2}{4\sigma^2} \right] \quad (5)$$

The incoming light is assumed to impinge upon the interface from medium 1 with a local incident angle θ_i equal to the local slope s , and a local transmission angle θ_t . The angle of transmittance with respect to the

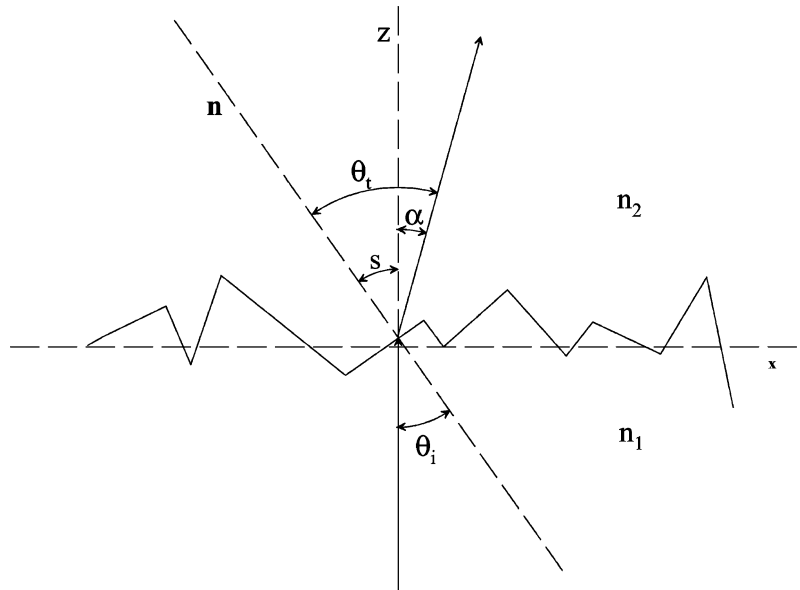


Figure 4 Schematic drawing of the parameters of the facet model [16].

average normal \mathbf{z} is α :

$$\begin{aligned} \alpha &= \theta_i - s & \text{for } n_1 > n_2 \\ \alpha &= \theta_i + s & \text{for } n_1 < n_2 \\ \alpha &= 0 & \text{for } n_1 = n_2 \end{aligned} \quad (6)$$

The local angle of incidence and transmittance are related through Snell's law:

$$n_1 \sin \theta_i = n_2 \sin \theta_t \quad (7)$$

The incident light is considered to be unpolarized. For a local angle of incidence $\theta_i = s$, the total transmittance $T(s)$ equals: $T(s) = 1/2[T_s(s) + T_p(s)]$, where T_s and T_p are the perpendicular- and parallel-polarization transmittances, respectively [15].

Of interest is the intensity of the transmitted light $\langle T_\alpha \rangle$ measured, for example, by a UV/vis spectrometer along the z -axis, far away from the surface, i.e., at small

values of α , for instance $\alpha \approx 1^\circ$:

$$\langle T_\alpha \rangle = \int_{s_1}^{s_2} T(s) P_s(s) ds \quad (8)$$

here s_1 and s_2 are the values of s that, through Equations 6 and 7, correspond to $\alpha = -1^\circ$ and $\alpha = 1^\circ$, respectively. In case of a smooth surface ($L/\sigma \approx 100$) or a small refractive index difference, most of the light will be transmitted into the forward direction along the z -axis at small values of α . If the surface is rough ($L/\sigma \approx 1$) and the refractive indices are not identical, then the light will be scattered over larger values of α . This is depicted in Fig. 5, which shows the transmittance in a small cone, defined by $\alpha \pm 1^\circ$, for different surface roughnesses. In reality, the cone dimensions are, of course, mainly determined by the diameter of the detector and the detector-surface distance. In Fig. 5, it can be seen that in case of a smooth surface ($L/\sigma = 100$), most of the light is scattered in the forward direction, even

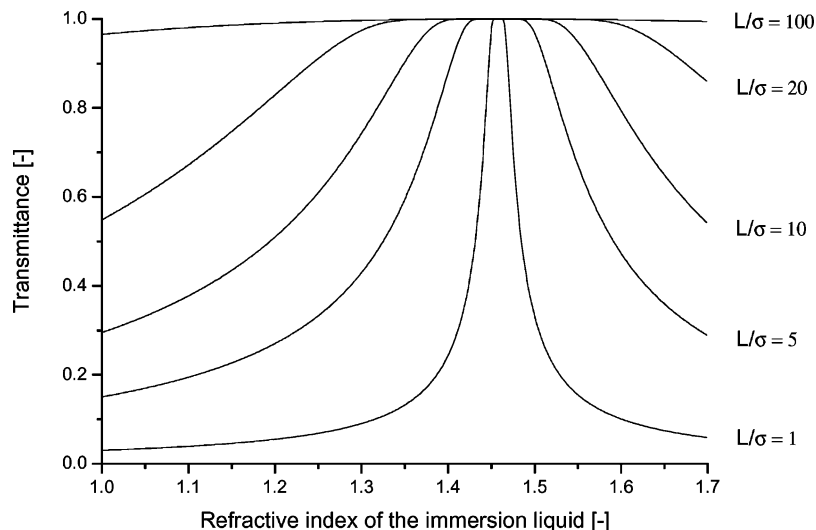


Figure 5 Calculated transmission of light passing a sheet with refractive index 1.458 and different roughnesses, using the facet model.

for relatively large refractive index differences. Consequently, the curve is nearly indistinguishable from that described by the flat surface expression (Equation 4 and Fig. 3). With increasing surface roughness, most of the light is scattered outside the small cone, except in the case of small refractive index differences. This leads to a favorable, much more pronounced maximum in the transmittance as a function of refractive index of the medium.

Although both the flat-surface expression (Equation 4) and the curves calculated with the simple facet model (Fig. 5) indicate a slightly asymmetric dependence of the transmission of the refractive index of the immersion liquids, the data points over all were represented best with symmetric equations, i.e. the Gauss Equation 9, the Lorentz Equation 10, or a polynomial fit (11):

$$y = y_0 + \frac{\sqrt{2}A}{w\sqrt{\pi}} e^{-2\frac{(x-x_c)^2}{xw^2}} \quad (9)$$

$$y = y_0 + \frac{2Aw}{\pi(4(x-x_c)^2 + w^2)} \quad (10)$$

$$y = y_0 + \frac{A}{1 + A_1\left(\frac{x-x_c}{w}\right)^2 + A_2\left(\frac{x-x_c}{w}\right)^4} \quad (11)$$

with y_0 the level of the baseline, A the area under the fitted curve, A_1 and A_2 empirical parameters yielding the best fit, w the full width at half maximum, and x_c the x values associated with the maximum y value. Since the transmission cannot be negative and the baseline should be at 0% transmission for large refractive index differences between the sample and the immersion liquid, the value 0% was selected for y_0 . In Fig. 6, it can be seen that the shapes of the Lorentz and the polynomial fit were essentially identical, but these two curves clearly differed from the Gauss fit; the polynomial and the Lorentz fits better matched the data points in the proximity of the transmission maximum than the Gauss approximation. In spite of this, all resulting

refractive indices at the transmission maxima (n_{Tmax}) were close together (1.4566, 1.4566, and 1.4525, respectively). The deviation of n_{Tmax} from the given value of quartz glass (1.458) amounted to 0.0014 for the Lorentz and the polynomial fit, and to 0.0055 from the Gauss fit. As mentioned above, the data are expected to follow a slightly asymmetric curve, and since the data points were fitted with symmetric functions, some deviation of the interpolated transmission maximum might arise as a consequence. The transmissions at n_{Tmax} in Fig. 6 reached 100.3 (Lorentz fit), 90.4 (Gauss fit) and 100.3% (polynomial fit), respectively; i.e., there was always some deviation from the ideal value of 100%, which we consider to be due to a non-perfect representation of the data points by the functions used. The value obtained from the Gauss fit deviated in a more pronounced manner from the ideal transmission maximum of 100% than those of the other two fits, which might be expected since the Gauss fit generally matched the data points around n_{Tmax} worse than the other two methods.

3.2. Experimental results

Quartz glass, crown glass and poly(vinylidene fluoride) (PVDF) were the materials used for the present experimental studies. In the case of PVDF and, of course, in general, immersion liquids evidently have to be selected which do not dissolve or swell the materials to be analyzed during the period required for the measurements.

The surfaces of the above materials were roughened as described in the experimental section and, subsequently, used for the determinations of their refractive index at 20°C. The samples were positioned in the liquid cell with their roughened surface perpendicular to the incident beam and immersed in different liquids with refractive indices between 1.36 and 1.62, and the UV/vis spectra recorded. Table I lists the immersion media referred to in this study and their refractive indices at 589 nm and 20°C, obtained from the respective suppliers, from the literature and from measurements carried out in this work. With few exceptions, the

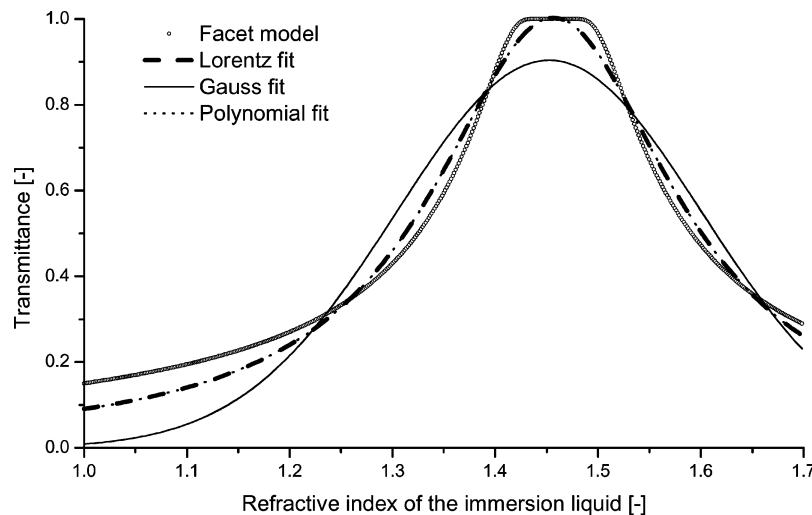


Figure 6 Transmitted light for a high-roughness surface ($L/\sigma = 5$) according to facet model fitted with Lorentz, Gauss or polynomial functions (Equations 9–11).

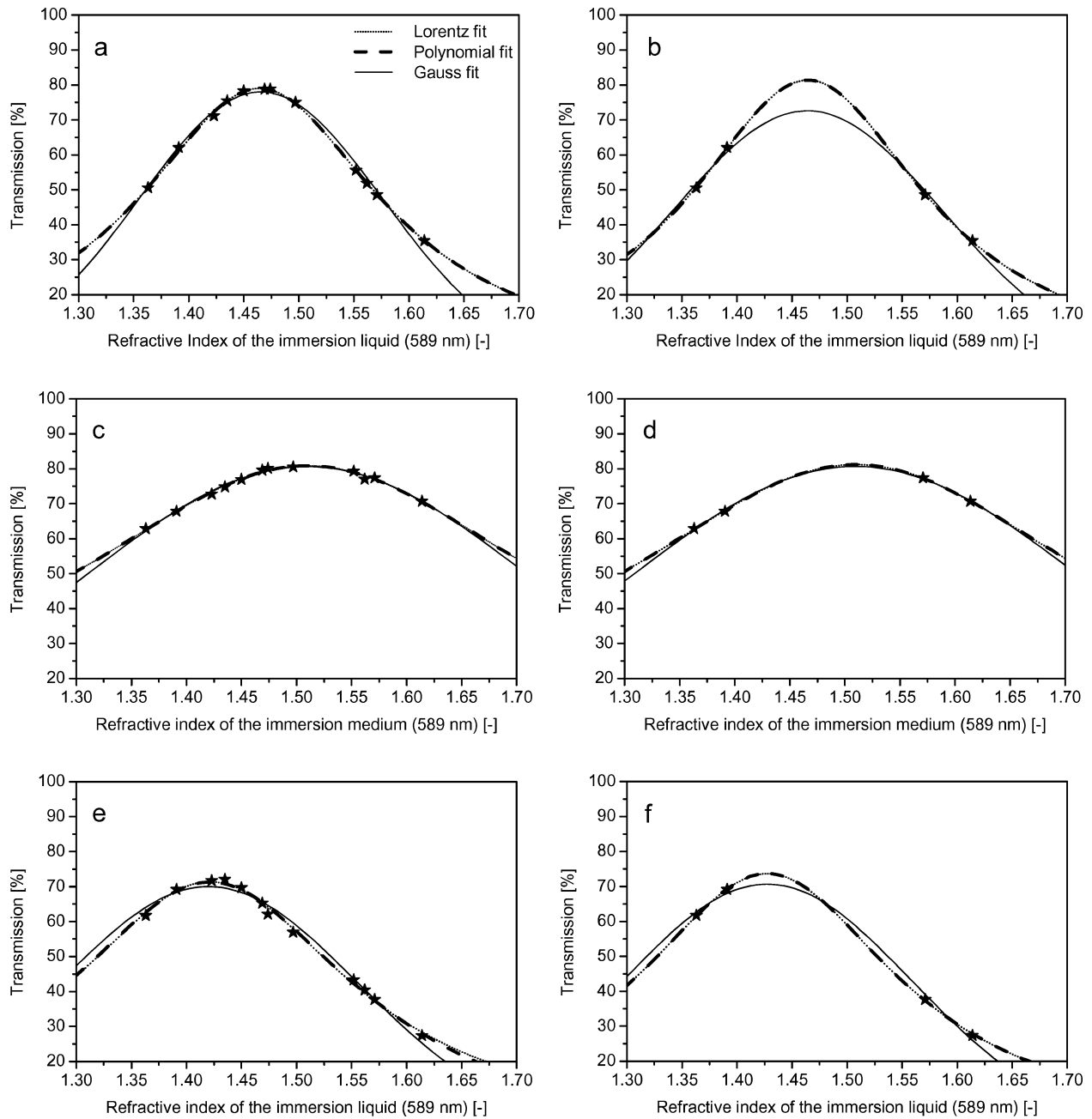


Figure 7 (a) Transmission of light passing a quartz glass sample immersed in various media. The 12 data points were fitted with various equations (Gauss, Lorentz and polynomial, see text). For the liquids used, see Table II. (b) Same, but with 4 liquids, see Table II, (c), (d) crown glass; (e), (f) PVDF.

refractive index values from the three sources were in agreement within about ± 0.001 . In the following, refractive indices from the suppliers were used.

The refractive indices reported below for the quartz, crown glass and PVDF films refer to the average values obtained for three different specimen. For comparison, the refractive indices of the studied materials were also measured by ellipsometry (of planar samples without scratches) and in addition compared to values reported in the literature. As indicated above, various selections of liquids (reference number in Table I) were used for the determination of the maximum transmission values, n_{Tmax} , i.e.

twelve liquids (2–13)

six liquids (3–5, 7, 9, 13)

the two liquids with the lowest and the two liquids with

the highest refractive indices (2, 3, 12, 13)

the two liquids with the refractive index with the lowest negative and the two liquids with the lowest positive difference to n_{Tmax} , i.e. 5–8 for quartz glass, 8–11 for crown glass and 2–5 for PVDF.

Data fits for quartz glass, crown glass and PVDF are shown in Figs 7a–f and the obtained n_{Tmax} values for the different sets of liquids and evaluation methods are collected in Table II. It is evident from the Figures that the Lorentz and the polynomial fits were close together and often virtually identical, whereas the Gauss fits in most cases clearly deviated from the other two fits. Also, as already indicated by the above model calculations, the Gauss fits matched the data points usually less precise than the Lorentz and polynomial fits (occasionally, though, similar matching was obtained for

TABLE II Refractive index at maximum transmission ($n_{T_{\max}}$) evaluated with different fit methods indicated and different sets of liquids

Sample	Liquids		$n_{T_{\max}}$			n	
	Table I (No.)		Lorentz	Polynomial	Gauss	Ellipsometry	Literature
Quartz glass	2-13	(12)	1.466	1.466	1.466	1.459	1.4584 [20, 21]
	3-5, 7, 9, 13	(6)	1.466	1.467	1.468		
	2, 3, 12, 13	(4)	1.467	1.467	1.467		
	5-8	(4)	1.463	1.463	1.463		
Crown glass	2-13	(12)	1.511	1.511	1.510	1.519	1.52 [22]
	3-5, 7, 9, 13	(6)	1.509	1.509	1.511		
	2, 3, 12, 13	(4)	1.510	1.512	1.510		
	8-11	(4)	1.513	1.514	1.513		
PVDF	2-13	(12)	1.422	1.422	1.422	1.415	1.42 [23]
	3-5, 7, 9, 13	(6)	1.422	1.423	1.424		
	2, 3, 12, 13	(4)	1.425	1.425	1.424		
	2-5	(4)	1.422	1.423	1.423		

all three fits). However, the $n_{T_{\max}}$ values obtained with the three fit methods were close together and differed at maximum by only 0.002. For a given fit method, $n_{T_{\max}}$ depended only moderately on the set of liquids used. For instance, the $n_{T_{\max}}$ values resulting from the evaluation with the 12 and 6 liquid set referred to above differed at most by 0.002. The largest difference was 0.003 when $n_{T_{\max}}$ obtained with the 12 liquid set was compared with the values resulting from the analysis with only 4 liquids.

No systematic dependence of the refractive indices obtained on the surface roughness was found (the sample roughnesses R_a were between 293 nm and 1399 nm, and R_q between 363 nm and 1917 nm; in addition, wave-features with amplitudes in the range of about 100 μm and a wavelength in the millimeter range were detected in the PVDF films, resulting from the relatively low rigidity of the PVDF films).

The $n_{T_{\max}}$ values obtained with all sets of liquids and evaluation methods were compared with the refractive indices of the corresponding materials measured by ellipsometry, or literature data (Table II). A maximum deviation of 0.009 was found for the materials examined. No systematic deviations of $n_{T_{\max}}$ from refractive indices reported in the literature or determined by ellipsometry were observed, and no preference or disqualification of a particular fit method resulted if the ellipsometric or literature values were considered as reference values.

In addition to the experiments summarized in Table II, $n_{T_{\max}}$ was also determined by interpolation with three liquids with lower and one with higher refractive index (and vice versa) than that of the immersed body. It was found that $n_{T_{\max}}$ also in these cases represented the refractive index of the immersed body within an accuracy of 0.010, provided that at least one of the refractive indices of the immersion liquids was substantially lower and one distinctly higher (at least 0.05) than that of the material tested. Determination of the refractive index of an immersed body by extrapolation was also investigated. In order to obtain values within an accuracy of 0.010, it was of importance that on the one hand the refractive indices of a part of the selected immersion liquids differed by more than ca. 0.05 from $n_{T_{\max}}$, while on the other hand at least one refractive in-

dex of an immersion liquid should not deviate by more than ca. 0.02 from $n_{T_{\max}}$.

4. Conclusions

Interpolation of the maximum transmission of light through a solid immersed in liquids of various refractive indices is a suited method for the determination of the refractive index of materials with irregular surfaces with an accuracy better than 0.01. The precision is limited by the fit method and not by experimental variables such as limits in the precision of the refractive index of the immersion liquids, in the measurement of the transmission, and/or the precision of sample placement in the cuvette. The accuracy might be improved if a fitting procedure existed that represents the data better than the methods employed here. The Lorentz, the polynomial and the Gauss fit used resulted in nearly the same refractive indices; however, the Lorentz and the polynomial fit appear to better match the data than the Gauss fit. Six liquids with distinct refractive indices seem to be sufficient for the determination of the solid's refractive index, and even four liquids appear to be satisfactory, if their refractive indices are not too close to that of the sample to be examined. Obviously, the refractive indices of the immersion liquids provided by the suppliers are of sufficient precision for the evaluation of the transmission maximum, and the measurements can be performed with little effort and a standard UV/vis spectrometer.

A major advantage of the presented method for refractive index determinations of solids is that the quality of the sample surfaces does not play a role—in fact rough is preferred—and compared to the classical immersion method, the modified procedure described here avoids the search for a refractive index-matching-liquid.

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