Colloid Polym Sci 273:1041–1048 (1995)

ORIGINAL CONTRIBUTION

D. Freitas Siqueira
D. W. Schubert
V. Erb
M. Stamm
J.P. Amato

Interface thickness of the incompatible polymer system PS/PnBMA as measured by neutron reflectometry and ellipsometry

Received: 10 May 1995
Accepted: 27 June 1995

Dr. M. Stamm (✉)
D.W. Schubert · V. Erb · D.F. Siqueira
Max-Planck-Institut für Polymerforschung
Postfach 31 48
55021 Mainz, Germany

J.P. Amato
Institut für Mikrotechnik Mainz GmbH
Postfach 24 40
55071 Mainz, Germany

Abstract The techniques of neutron reflectometry and spectroscopic ellipsometry are compared as methods to measure the interface width between immiscible polymers. The interface thickness of the incompatible polymer system of polystyrene (PS) and poly (*n*-butyl methacrylate) (PnBMA) is determined by neutron reflectometry to (6.4 ± 0.2) nm and (8.6 ± 0.2) nm at temperatures of 120 and 156 °C, respectively. Some emphasis is put on the measurement of those values also by spectroscopic ellipsometry using the same materials. A special sample geometry is chosen for ellipsometric measurements to compensate for thickness changes of films during annealing, and the dispersions of PS and PnBMA films are determined. With respect to the determination of the interface widths, however, it turns out that in the available wavelength range of 280 to 700 nm spectroscopic ellipsometry is not sensitive enough to measure the thin interface width between PS and PnBMA films. Neutron reflectivity results obtained for PS/PnBMA are discussed with respect to the Flory–Huggins segment interaction parameter χ calculated within the approximations of mean-field theory.

Key words Polymer blends – interfaces – interdiffusion – ellipsometry – neutron reflectometry

Introduction

The miscibility between two polymers can be evaluated through the interfacial thickness. Generally, when two polymer films are put together and are heated above their glass transition temperature, a broad interface will develop with time in miscible systems, whereas the formation of a thin interface is characteristic of immiscible systems. The interfacial thickness may be taken as a measure of compatibility. For immiscible systems, both mean-field [1] and lattice [2] theories predict that some interdiffusion of polymer segments occurs at the interface to minimize the interfacial energy. It is proposed that the thickness of this interface is proportional to $\chi^{-0.5}$, while the interfacial tension between both polymers is proportional to $\chi^{0.5}$, where χ denotes the Flory-Huggins segment interaction parameter. Immiscible polymers have large and positive χ, the interfacial thickness is consequently very thin and the interfacial tension very high.

From mean-field theory [1], a relation between interfacial thickness, a_i, and χ for strongly immiscible polymers in the limit of infinite molecular weight is derived:

$$\frac{a_i}{2} = \frac{b}{\sqrt{6\chi}} \quad (\chi \gg \chi_S)\,, \tag{1}$$

where b is the mean Kuhn segment length and χ_S the value of χ at the spinodal. a_i is defined in the usual way by the tangent to the profile at $z = 0$ (see Eq. (2) below).

Colloid & Polymer Science, Vol. 273, No. 11 (1995)

On the basis of Eq. (1), a_i may be obtained when χ is known or viceversa. It should be kept, however, in mind that Eq. (1) is derived in the limit of $\chi \gg \chi_S$, neglecting differences in specific volume of monomers or concentration effects, for instance. It is thus not a very good approximation in the vicinity of the critical point or for materials with very different densities. Equation (1) similarly does not take into account changes of the chain conformation in the vicinity of the interface, enrichment of chain ends at the interface or fluctuation effects, which are discussed at the free surface of a polymer melt [3, 4] and are also expected at an interface between two immiscible polymers [5, 6].

Accordingly, a volume fraction profile is obtained [1–3]

$$\phi = \frac{1}{2}\left[1 + \tanh\left(2\frac{z}{a_i}\right)\right], \quad (2)$$

where $z = 0$ is defined as the midpoint of the interface between the two polymers. This profile is symmetric around $z = 0$ and similar restrictions hold as have been discussed for Eq. (1).

Measuring the interfacial thickness or the segment interaction parameter χ in immiscible systems is not a trivial task. Most common experimental techniques for the determination of the interface width between polymers have recently been compared [7, 8]. Neutron reflectometry is one of the most accurate methods to measure in particular the interface width and profile, if one of the polymers is deuterated [7–12]. In specific cases also x-ray reflectometry may be used to investigate the interface between immiscible polymers [11-13], when a suitable electron density contrast between the components is present. Ellipsometry similarly has proved to be an adequate method to measure the interfacial thickness [14–19], when a sufficient contrast in the index of refraction between materials is present. With ellipsometry there is nevertheless a principal problem for the determination of the interface width of polymers, when the interface width is small compared to the wavelength of light.

The purpose of this work is to compare the efficiency of spectroscopic ellipsometry (SE) and neutron reflectometry (NR) as methods to determine the interfacial thickness between two particular immiscible polymers. Neutron reflectometry (NR) measurements are presented for the system deuterated poly(styrene) (PS(D))/poly(*n*-butyl methacrylate) (PnBMA). The NR results and those obtained from SE measurements for this system are compared with respect to the available information to be obtained for interfacial thickness and details of the interface profile.

Experimental

Materials

Poly(*n*-butyl methacrylate) (PnBMA) was kindly supplied by Dr. Siol, Röhm GmbH, Darmstadt. Deuterated polystyrene (PS(D)) was synthesized at Max Planck Institut für Polymerforschung, Mainz. Molecular characteristics like molecular weights (M_w) and polydispersities (M_w/M_n) were obtained for PS(D) and PnBMA by gel permeation chromatography in tetrahydrofurane (THF). Data were calibrated with PS and PMMA standards, respectively. Glass transition temperatures, T_g, were measured by DSC at a heating rate of 20.0 °C min^{-1}. Data are summarized in Table 1.

Ellipsometry

The principle of ellipsometry is based on the fact that a monochromatic electromagnetic wave changes its state of polarization after striking a reflecting surface. This change is a function of the optical parameters of the whole system as shown in the basic equation of ellipsometry [20]:

$$\exp(i\Delta)\tan\Psi = f(n_k, d_k, \lambda, \phi), \quad (3)$$

where $\Delta(\Delta = 2p + 90°)$ and $\Psi(\Psi = a)$ are the ellipsometric angles obtained from the polarizer p and analyzer a settings with respect to the plane of incidence, n_k and d_k are the indices of refraction and thicknesses of each layer present in the system, λ is the wavelength, and ϕ the angle of incidence.

Spectroscopic Ellipsometry (SE) measurements were performed at room temperature in a Sopra Spectroscopic Ellipsometer ES 4G, with angle of incidence fixed at 75° and in a wavelength range from 280 to 700 nm. The measurements were taken at intervals of 5 nm. The measured and fitted curves were analyzed by means of the software Sopra Elli45. The interpretation of the multilayer system with layers for substrate/PnBMA/interface/PS(D) requires a model, which involves the dispersions for substrate, PnBMA and PS(D). We assume the refractive index of the unknown interface to be the mean value between the refractive indices of the PS(D) and PnBMA layers. The dispersions of substrate, pure PnBMA and pure PS(D) are

Table 1 Molecular characteristics of investigated materials

Sample	M_w (g/mol)	M_w/M_n	T_g (°C)
PS(D)	720000	1.13	102
PnBMA	150000	1.03	23

measured prior to the measurements at the interface or are taken from the literature if available.

Null-ellipsometry (NE) measurements in particular for the determination of film thickness were performed for comparison at room temperature in a self-built null-ellipsometer with angle of incidence fixed at 70° and at a wavelength of 632.8 nm [28].

Neutron Reflectometry (NR)

NR experiments are performed at the reflectometer TOR-EMA II at GKSS, Geesthacht. The wavelength of neutrons is fixed at $\lambda = 0.43$ nm, and reflectivities in the range 1 to 10^{-4} are measured with a position sensitive detector. Experimental resolution is taken into account using a convolution procedure for data analysis. The set-up is quite similar to the one described elsewhere [21]. The magnitude of the scattering vector in z-direction, k, is defined by $k = 2\pi/\lambda \sin\theta$, were θ is the angle of incidence.

Sample preparation for the ellipsometric measurements

Double films of PS(D) /PnBMA are prepared in the following way: a film of PnBMA ca. 40-nm-thick is spin-coated from solutions in toluene at the concentration of 5 mg/ml on a silicon wafer with a typical size of 7×2 cm^2. Silicon wafers kindly supplied by Wacker Chemitronics, Burghausen, were used as substrates. They are composed of Si with a top layer of natural SiO_2 typically 2-nm-thick. One-third of the spin coated film is again dissolved in toluene, building a step in the sample.

The thickness and dispersion of the PnBMA film are determined by SE (Fig. 1), following the Sellmeyer relation [22]. A PS(D) film ca. 170-nm-thick is spin-coated on float glass from a solution in toluene at the concentration of 20 mg/ml and floated off with distilled water. The floated PS(D) film is carefully picked up and arranged in such a way that one half covers the PnBMA film and the other half the SiO_2 surface, as shown in Fig. 2. The dispersion of PS(D) is obtained (Fig. 1) by measuring the region covered just by PS(D) (region 2 in Fig. 2). The whole system is kept at room temperature under vacuum during 24 h in order to eliminate remaining traces of water.

The surface roughness is measured by phase-measurement interference microscopy (PMIM) (Zygo Maxim 3D) [7] to be typically 1.2 nm. The multilayer system is first investigated by SE or NE at 20.0 °C in the different regions (Fig. 2) and then heated under vacuum at 140 °C for 2 h. After heating, the samples are quenched to 20.0 °C and ellipsometric measurements are again performed in the PnBMA, PS(D) and interface regions (regions 1, 2 and 3 in Fig. 2). A change in the thickness of the pure polymer films of about 0.3 nm is observed. This seems to be not a very significant change, because it lies in the accuracy limit of this method, while it causes, however, a change of ellipsometric angles of the same order of magnitude as expected from the interdiffusion at the interface (see discussion below). Thicker films can show changes up to 2.0 nm in the thickness after annealing, which makes control in the thickness of the pure polymer films after heating above T_g quite important.

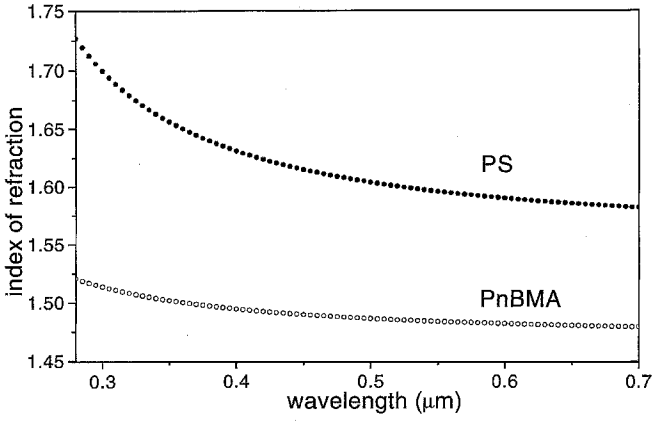

Fig. 1 Dispersion of index of refraction PS(D) and PnBMA measured by spectroscopic ellipsometry in the wavelength range of 280 to 700 nm at room temperature

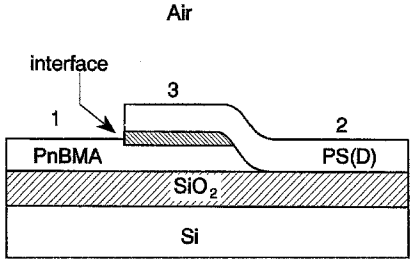

Fig. 2 Schematics of multilayer sample for ellipsometric measurements. Different positions 1, 2 and 3 for measurements of PnBMA, PS(D) and double films are indicated

Sample preparation for NR

Thin films for neutron reflectometry experiments are prepared by first spincoating separate films of PS(D) and PnBMA on float glass substrates from toluene solutions (concentrations of 20 mg/ml and 10 mg/ml, respectively). PS(D) is the top layer and is floated off on water. It is then deposited on a bottom layer of PnBMA, which is directly spin-coated on a float glass substrate. The sample size is

$10 \times 10\ cm^2$ and homogeneity over this area is checked by PMIM. A typical surface roughness is 1.0 nm. The thickness of individual films is of the order of 100 nm, which is obtained accurately from x-ray reflectometry experiments. The samples are annealed under vacuum in the range of 120 to 156 °C, respectively, for 2 h and then quenched to room temperature, where the NR experiments are performed. Sample preparation is thus largely identical to the one used for ellipsometry.

Results and discussion

Neutron reflectometry

Neutron reflectometry is a technique which can be applied when a suitable neutron contrast is present at the interface between materials. This is achieved in our case by the use of deuterated poly(styrene). Reflectivity curves obtained from a PS(D) /PnBMA double-layer system on glass after annealing at 120 and 156 °C for two hours are shown in Fig. 3. Using the matrix technique [23] model fits are performed which are indicated by the solid lines in Fig. 3. For the interfaces air/polymer and polymer/glass error functions are used for the scattering density profiles, where the interface widths are characterized by the variances σ of those distributions. The interface between the two polymers is described with a profile for the scattering density according to Eq. (2), where the characteristic parameter for the interface width is a_i. Parameters obtained from the fit are: a_i, film thicknesses $d_{PS} = 167.8$ nm, $d_{PnBMA} = 86.3$ nm and interface widths $\sigma_{glass-PnBMA} = 0.9$ nm. and $\sigma_{PS-air} = 0.9$ nm. Scattering densities correspond to usual densities of materials. Those parameters are obtained largely independent from each other, when the particular layer model for scattering density is assumed [10, 11]. From the model fit, thus the interfacial thickness a_i is obtained to be in the range of (6.4 ± 0.2) nm to (8.6 ± 0.2) nm, corresponding to the temperature range of 120 to 156 °C, respectively.

Using these results for the interfacial thickness, the Flory–Huggins interaction parameter χ can be calculated following a more detailed discussion according to Schubert and co-workers [24]. χ turns out to be in the range 0.0093 to 0.0055 at the investigated temperatures. Approximations as discussed with respect to Eq. (1) are used. This means in particular that fluctuation effects at the interface and the distortion of the chain conformation at the interface are not taken into account. Both effects may cause a broadening of the interface as compared to the simple mean field picture. The χ parameter thus can be expected to be too small, when calculated on the basis of Eq. (1). The calculated values of χ are, on the other hand,

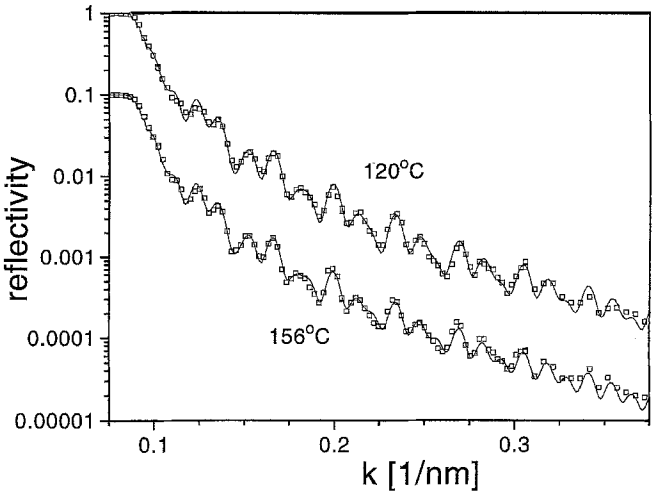

Fig. 3 Neutron reflectivity curves of annealed samples (120 and 156 °C for 2 h) with a PS(D)-film deposited on PnBMA on a glass substrate. Solid lines correspond to fits to experimental data as explained in the text

within a factor of 2 quite comparable to interaction parameters determined by small-angle neutron scattering from PS and PMMA diblock copolymers in a similar temperature range [25]. Miscibility of PS and PnBMA at lower molecular weights or higher temperatures may be predicted [24] on the basis of the measured χ parameters, which has recently also been observed experimentally [26].

With respect to the comparison of NR with ellipsometry, it should be emphasized, however, that the NR technique is well capable to measure the narrow interfaces between incompatible polymer materials, if one component is deuterated. Parameters like indices of refraction, individual film thicknesses, surface and interface roughnesses and the interface width between the polymers can, to some extent be determined independently from each other [10, 11], focusing on different aspects of the reflectivity curve. The exact form of the profile between the polymers will be quite difficult to determine. Ambiguities of the technique can be overcome by additional measurements of film thicknesses, indices of refraction and surface roughnesses of individual films prior to the preparation of the multilayer sample, and most parameters thus will have to be only slightly refined, leaving as the only main fit parameter the interface width a_i between the polymers. This parameter then can be determined with good precision.

Ellipsometry

The dispersions of PS(D) and PnBMA pure films are necessary to model the SE data for the multilayer system.

From Ψ and Δ measurements for a pure film of known thickness the values of $n(\lambda)$ were obtained. These values can be fitted following the Sellmeier relation [22]:

$$n^2 = 1 + \frac{e\lambda^2}{\lambda^2 - f^2}, \tag{4}$$

where e and f are characteristic parameters of the materials.

To our knowledge, there is only little information about wavelength dispersion of polymers in the literature [18, 19]. The values of refractive indices obtained for PS(D) (Fig. 1) range from $n = 1.7267$ at $\lambda = 280$ nm to $n = 1.5817$ at $\lambda = 700$ nm. The fit parameters obtained for PS are $e = 1.4361$ and $f = 145.91$ nm. They are in good agreement with those measured by Sauer and Walsh [18, 19]. For PnBMA the index of refraction ranges from $n = 1.5205$ at $\lambda = 280$ nm to $n = 1.4790$ at $\lambda = 700$ nm (Fig. 1). The fit parameters found for PnBMA are $e = 1.1678$ and $f = 100.34$ nm. The fitted curves are analyzed by means of a software, where e and f vary following the Sellmeier relation, and from this variation $n(\lambda)$ is obtained. Then, the ellipsometric angles are calculated and compared to the ellipsometric angles which are measured. The variation in e, f, and thickness is made until a minimum difference between the calculated and measured ellipsometric angles is found. This difference is calculated by the method of root mean square superposition and a standard deviation is obtained. For the pure films of PS(D) and PnBMA the standard deviation values are of the order of 0.005. In the range of wavelength of 280 to 700 nm the values of the extinction coefficient κ (the imaginary component of the complex index of refraction) for PS(D) and PnBMA are close to zero and are neglected here. For the Si-wafers, we use literature data for n and κ [27], which agree with our experimental results.

Using the dispersion files the thicknesses of the PS(D) and PnBMA films alone are obtained to be 167.1 nm and 43.5 nm, respectively (region 2 and 1 in Fig. 2). The thicknesses of the same PS(D) and PnBMA films, however, arranged on top of each other (region 3 in Fig. 2) were determined from a model fit to the Δ and Ψ values (see Fig. 4) to 166.6 nm and 43.2 nm, respectively. Thickness variations at different locations of the films are of the order of 1 nm, as measured also by NE. The solid curves in Fig. 4 represent the experimental data, while the dashed curves represent the best fits. The fit is performed in the whole available wavelength range (280 to 700 nm), while for a better comparison in Fig. 4 only the range 280 to 400 nm is shown. In this range, one would also expect the most significant changes in ellipsometric angles during interdiffusion, since the difference of the indices of refraction between the two polymers is largest at those wavelengths.

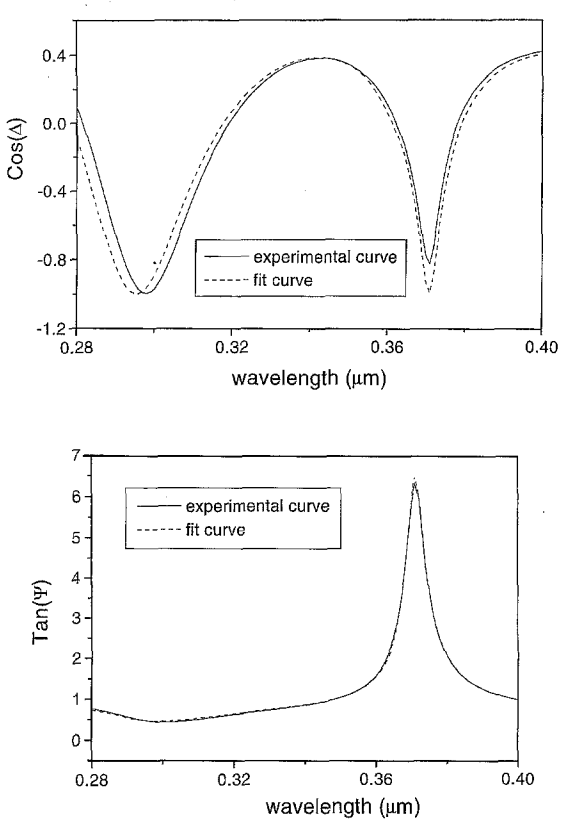

Fig. 4 Ellipsometric angles Δ and Ψ as a function of wavelength λ measured for a 166.6-nm-thick PS(D) film arranged on a 43.2-nm-thick PnBMA film prior to annealing. The solid line represents the experimental curve and the dashed line the best fit, as explained in the text

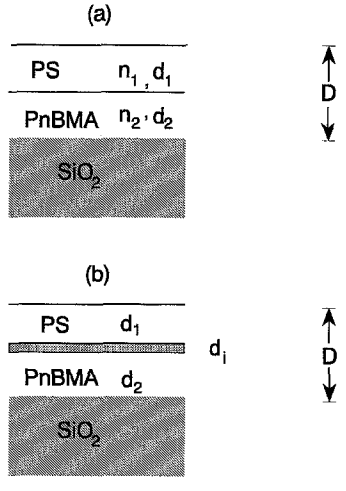

Fig. 5 Multilayer model used for the computer calculations (a) before annealing, in the absence of an interface and (b) after annealing, with a homogeneous interface layer of thickness d_i. The thicknesses of the pure PS and PnBMA films are d_1 and d_2, respectively, and the total film thickness is $D = d_1 + d_2 + d_i$

A layer model as indicated in Fig. 5a is used for the fit, where the layer thicknesses d_1 and d_2 are the only parameters for the fit. D is the total film thickness and is calculated from d_1 and d_2. A comparison of the different

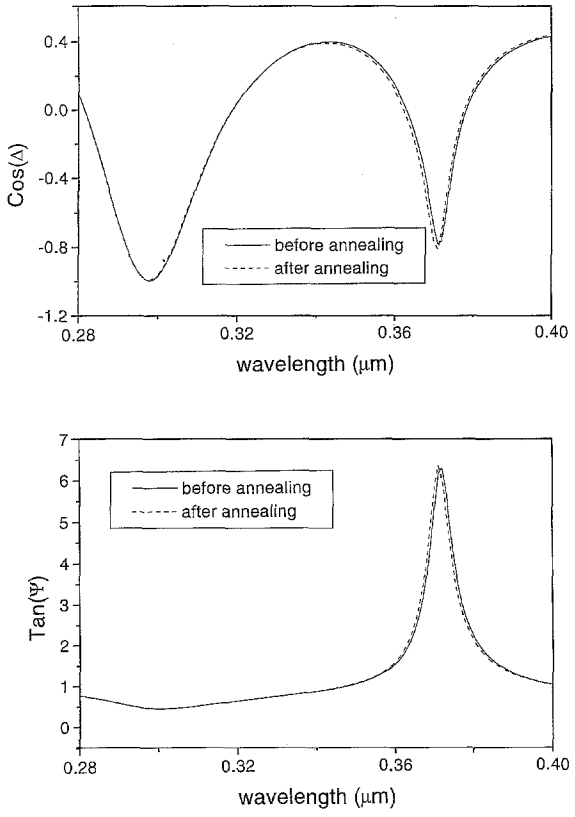

Fig. 6 Ellipsometric angles Δ and Ψ measured for the double layer film system (Fig. 4) before (solid curve) and after annealing (dashed curve) as a function of wavelength λ

film thicknesses in different regions (for the PS film regions 2 and 3, for the PnBMA film regions 2 and 1 in Fig. 2) shows that the thicknesses of individual films in different regions are obtained consistently and deviations are not larger than 0.5 nm. The experimental $\tan\Psi$ curve of the layer (Fig. 4) is quite well fitted over the entire wavelength range, while larger deviations are observed between the experimental and fitted curves for $\cos\Delta$. The standard deviation for the fit is 0.03. For our system $\cos\Delta$ is more sensitive on details of the model than $\tan\Psi$. Deviations might be due to limitations of the model, lateral sample inhomogeneities, film anisotropy, interface roughness or traces of impurities. The simple layer model for the fit would have to be modified to obtain a better fit.

After annealing the sample at 140 °C for 2 h new ellipsometric measurements were performed. The data points are slightly shifted in relation to those obtained before annealing, as shown in Fig. 6. The shift is most pronounced in the wavelength range of 280 to 400 nm, where the difference between the indices of refraction of PS(D) and PnBMA is largest. The shift observed between the $\cos\Delta$ curves before and after annealing is not very large, which becomes particularly evident when it is compared to the accuracy of the fit. Thus, the deviations obtained between experimental data and the calculated curve from the fit (Fig. 4) are of the same order of magnitude or even larger than the observed changes by annealing. On the other hand, for the $\tan\Psi$ curves the opposite is the case, and the shift observed before and after sample annealing is larger than the deviations observed in the fit. One might therefore be tempted to interpret those differences in terms of interface formation between PS and PnBMA. This, however, proves to be quite dangerous, as will be discussed below.

In order to verify whether the observed changes in $\cos\Delta$ and $\tan\Psi$ are connected with a change in the interfacial thickness d_i, calculations were performed considering a multilayer model with three values for the interface thickness: $d_i = 0$ nm, 5 nm and 10 nm (Fig. 5b). The values of the individual film thicknesses of PS(D) and PnBMA are taken from measurements from region 3 (see Fig. 2) to be 166.3 and 43.0 nm, respectively, and correspond with only small deviations to the unannealed ones. Values are corrected for a change of thickness of about 0.3 nm after annealing, which corresponds to the change obtained from measurements in regions 1 and 2 before and after annealing. The thicknesses of the initial pure polymer layers of PS(D) and PnBMA in the layer system, d_1 and d_2 in Fig. 5a, are, however, for the model calculations reduced by $d_i/2$, i.e., 2.5 nm and 5 nm, respectively, for the interfacial thicknesses of $d_i = 5$ nm and 10 nm (Fig. 5b), thus keeping the total film thickness D constant, when the interfacial layer is introduced. The refractive index of the interface is assumed to be the mean value between the refractive indices of PS(D) and PnBMA. The resulting calculated curves considering interface thicknesses $d_i = 0$ nm, 5 nm and 10 nm are nearly indistinguishable. They fit very well the experimental $\tan\Psi$ data, as shown in Fig. 7 for $a_i = 0$ and 10 nm. On the other hand, the calculated curves show significant deviations with the experimental $\cos\Delta$ curve, while calculated $\cos\Delta$ curves are, however, all the same for 0 nm, 5 nm and 10 nm interface thickness. The values of the standard deviations are consistently of the order of 0.04. This clearly indicates that the observed shifts in $\cos\Delta$ and $\tan\Psi$ for the system PS(D) and PnBMA before and after annealing (Fig. 6) do not provide sufficient information about the interfacial thickness between the polymers. Under the employed conditions, spectroscopic ellipsometry proves to be fairly insensitive to changes of the interfacial thickness d_i between the two polymers. It has been noted already previously [23], in particular for the case when the interface width is smaller than the wavelength of light, that ellipsometry is not sensitive to determine the interfacial thickness.

It should be noted that ellipsometry is also expected to be insensitive to details of the model profile [28], which

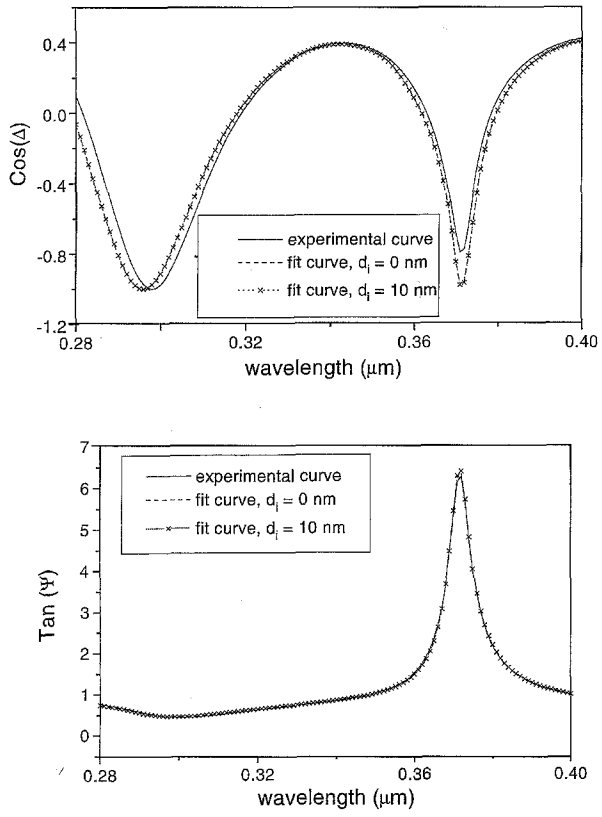

Fig. 7 Ellipsometric angles Δ and Ψ measured for the double layer film after annealing. The solid line corresponds to the experimental data, the dashed lines are the fit curves considering interface thicknesses of $d_i = 0$ nm and $d_i = 10$ nm

again has been checked by us by additional calculations. Thus at a wavelength of 632.8 nm virtually no detectable changes in Δ and Ψ for NE are obtained changing between a single step profile, a linear profile, or a hyperbolic tangent profile. By similar calculations a conversion factor between d_i and a_i of 5.7 is calculated, meaning that, for instance, an interface width of $d_i = 5$ nm (single step profile) corresponds to a value of $a_i = 29$ nm (hyperbolic tangent profile). Other calculations indicate that neither a change of the angle of incidence nor variations in film thicknesses provide a significant improvement of the situation. One has to keep in mind, however, that there are several systematic differences between SE and NE caused by different experimental procedures of data acquisition and detection, which makes a direct comparison of the two techniques difficult.

As a result of those calculations, we were not able to find conditions where ellipsometry is sensitive enough to allow the determination of the interface width between PS and PnBMA. Other factors like changes in film thickness during annealing, sample inhomogeneity, roughness, film anisotropy, impurities, etc., which we were not able to control in a sufficient way, cause much larger changes of ellipsometric parameters than does the expected change of the interface width.

To illustrate this point further, for example, a small change in the total film thickness might be considered. As described in the experimental part, with the help of the particular multilayer sample with different regions (Fig. 2), it is possible to control the occurrence of larger changes in the pure PS and PnBMA film thicknesses during annealing. Nevertheless, there might still be a relatively small change in the total layer thickness D after annealing (see Fig. 5b), which will be difficult to quantify by ellipsometry and which we may assume for a model calculation to be of the order of 0.5 nm only, i.e., less than 0.3% of the total film thickness. The ellipsometric angles Δ and Ψ (at $\lambda = 633$ nm) corresponding to this situation change quite significantly (Δ from 260.52° to 261.04° and Ψ from 19.88° to 20.04°), which becomes evident when those changes are compared to the changes expected for the formation of an interface (Δ from 260.52° to 260.51° and Ψ from 19.879° to 19.881° for a change of $d_i = 0$ to 5 nm). Such a small thickness variation is well in the range experimentally observed for pure PS and PnBMA films and can easily be explained by stress relaxation. It is important to emphasize that during the experiment it will be hard to judge whether a measured change of ellipsometric angles in this range will be due to interface formation, or whether it will be due to a small decrease in the total film thickness. The film thickness variation influences ellipsometric data thus much more dramatically than does interfacial diffusion.

The determination of the thin interface width between the investigated polymers by ellipsometry thus proves to be difficult. The general application of the technique, however, depends very much on the parameters of the particular system and the situation may be much more favorable for other polymers, in particular when the difference in the indices of refraction is larger and interfaces are broader.

Conclusions

We have performed a comparison of neutron reflectometry and ellipsometry for the determination of interface widths in incompatible polymer systems. For the particular system investigated, PS and PnBMA, the interface width is determined by neutron reflectometry to be 6.4 ± 0.2 nm and 8.6 ± 0.2 nm at 120 and 156 °C, respectively. From spectroscopic ellipsometry the dispersions for the pure polymers are presented, however, a reasonable value for the interfacial thickness cannot be given by spectroscopic ellipsometry in the wavelength range of 280 to 700 nm. This is due to the fact that the ellipsometric angles are not

sensitive enough to the small changes in interface width in this wave length range. Neglecting fluctuation effects or conformational changes at the interface, from the measured interface width the Flory–Huggins interaction parameter χ is obtained to be 0.0093 and 0.0055 at 120 and 156 °C, respectively. Neutron reflectometry thus can provide a measure of the interaction parameter χ of incompatible polymer systems, which is quite difficult to obtain otherwise, through the determination of the interface width in the framework of mean field theoretical predictions.

For the investigation of incompatible systems, where the interface widths are generally quite small, ellipsometry is not sensitive to details of the profile of the layer system and care must be taken to account, for instance, for the change of sample thickness during annealing. It is, however, still a very helpful and relatively simple technique when profiles are broad and refractive index contrasts are large. Neutron reflectometry, on the other hand, can provide in this range of interfacial thicknesses (e.g., 2–10 nm) very reliable results, when one component is deuterated. Also the form of the profile might be obtained. Neutron reflectivity thus clearly has some advantages over ellipsometry, when the interfaces between incompatible polymer systems are going to be determined, which is mostly due to the favorable contrast situation when deuterated components are used.

Acknowledgements We acknowledge interesting discussions with Dr. B. Sauer, technical help of B. Derichs during the neutron experiments, and of Th. Wagner during sample preparation. Neutron reflectivity experiments were performed at GKSS Geesthacht within a collaboration agreement with Max Planck Institut für Polymerforschung and were financially supported by BMFT.

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