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High-Refractive-Index Thin Films Prepared from Trialkoxysilane-Capped Poly(methyl methacrylate)−Titania Materials

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High-refractive-index trialkoxysilane-capped PMMA−titania hybrid optical thin films were prepared by an in situ sol−gel process, followed by spin-coating and multistep baking. The acrylic monomers used were methyl methacrylate (MMA) and 3-(trimethoxysilyl)propyl methacrylate (MSMA). Titanium(IV) *n*-butoxide was used for the preparation of the titania network. By using catalyst-free polymerization and adjusting the solvent THF amount for polymerization, the titania content in the hybrid thin films can be as high as 70.7 wt %. The FTIR and DSC results indicate the successful bonding between the organic and inorganic moieties and the good dispersion of the PMMA segment in the titania network. The AFM study demonstrates that the prepared hybrid thin films have excellent planarization. The FE-SEM results suggest that the titania moieties in the prepared hybrid materials **T10**−**T80** are on the nanosize scale. The dispersion of the refractive index and extinction coefficient in the wavelength range of 190−900 nm was studied. The off-resonant refractive indices of the prepared hybrid thin films **T10**−**T90** at 633 nm were in the range of 1.505−1.867 as the titania content linearly increased from 2.9 to 70.7 wt %. However, the Abbe number exhibits the reverse trend. The shift of the absorption maximum of the prepared hybrid thin films can be correlated with the titania content. The prepared hybrid films show potential applications in optical devices, which exhibit very high optical transparence in the visible region.

Introduction

Research interest in organic−inorganic hybrid materials remains very high.[1−4] The sol−gel technique has been shown to produce organic−inorganic materials with novel physical and chemical properties.[1−2] Applications of hybrid materials in optoelectronic devices have been extensively reported, such as contact lenses,[5] high-refractive-index materials,[6,7] waveguide materials,[8] optical coatings,[9] nonlinear optical materials,[10] photochromic materials,[11] and optical devices.[12]

Organic−inorganic hybrid materials can be potential candidates for high-refractive-index materials. Wilkes and co-workers successfully prepared triethoxysilane-capped polymer−titania hybrid materials, including poly(arylene ether ketone) (PEK),[6] and poly(arylene ether sulfone) (PES).[6] The refractive indices and the Abbe numbers of the PES−TiO_2 and PEK−TiO_2 hybrid materials, depending on the TiO_2 content, were in the ranges of 1.60−1.75 and 20−30, respectively. Wilkes and co-workers also prepared poly(arylene ether phosphine oxide)−titania hybrid materials,[13] which showed properties similar to those of PES−TiO_2 and PEK−TiO_2. Poly(methyl methacrylate) (PMMA) has been recognized as an excellent optical polymer for use in optical fibers, optical disks, and lenses.[14] However, the refractive index of PMMA is limited to 1.49. Hence, it will be interesting to prepare high-refractive-index polymers based on PMMA−titania materials. Zhang et al. successfully prepared poly(methyl methacrylate)−titania hybrid materials using the chelating ligand allyl acetylacetone as a coupling agent.[15] However, the titania content in the prepared materials was limited, and large amounts of solvent and chelating agent remained in the materials. Hybrid thin film properties were not investigated in their study. Our laboratory has successfully prepared

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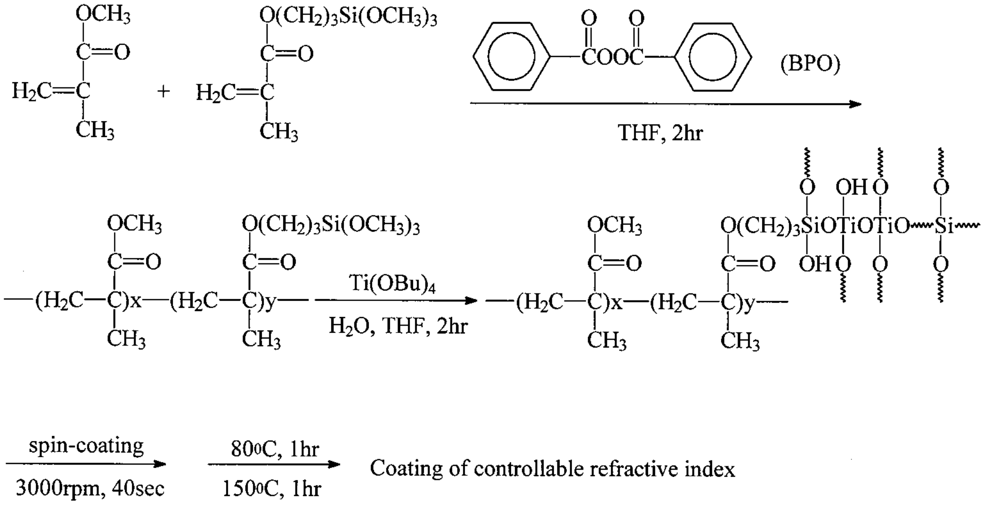

Figure 1. Reaction scheme for the preparation of the high-refractive-index trialkoxysilane-capped poly(methyl methacrylate)–titania films **T40**–**T90**.

trialkoxysilane-capped PMMA–titania hybrid optical thin films by the sol–gel technique.[16] The refractive indices of the prepared hybrid thin films were in the range of 1.505–1.553, with Abbe numbers larger than 30. However, the titania content in the prepared films was limited to 11.7 wt %. If the titania content was more than 11.7 wt %, the polymerization solution was easily gelled before spin-coating into a thin film. This was probably due to the fast reaction of $Ti(OBu)_4$ because of the acid catalyst and insufficient polymerization solvent. Furthermore, the domain size of the titania in the hybrid materials was difficult to detect, as noted in our last publication.[16]

It has been reported that the hydrolysis and condensation reaction can still be performed via acid-free polymerization for the case of $Ti(OR)_4$.[17] Hence, the acid-catalyst-free polymerization for the hybrid materials might provide a new reaction scheme for the preparation of trialkoxysilane-capped PMMA–titania hybrid optical thin films with high titania contents. In this study, a modification of the previous reaction scheme[16] was used to prepare high-refractive-index thin films **T40**–**T90** based on trialkoxysilane-capped PMMA–titania materials. Figure 1 shows the current reaction scheme. The acrylic monomers used were methyl methacrylate (MMA) and 3-(trimethoxysilyl)propyl methacrylate (MSMA). The alkoxide monomer for the preparation of the titania segment was titanium *n*-butoxide. MMA and MSMA was prepolymerized in THF first and then $Ti(OBu)_4$, and H_2O in THF was added into the reaction mixture. The reaction mixture was spin-coated as a thin film before gelation. Then, it was baked in two stages of curing temperatures to finish the polymerization. No acid catalyst was added to the polymerization solution, and the amount of THF solvent was adjusted to avoid gelation. The properties of the newly prepared hybrid materials **T40**–**T90** were compared with those of previously prepared **T10**–**T33**. The structures, thermal properties, AFM and FE-SEM morphologies, and dispersion of the refractive indices and extinction coefficients in the wavelength range of 190–900 nm were examined in the present study. The effects of the titania content on the properties of the hybrid materials were investigated.

Table 1. Monomer Mixtures (wt %) for Preparing Hibrid Thin Films T40–T90[a]

	T40	T50	T55	T60	T65	T70	T80	T90
MMA	32.8	27.4	24.6	21.9	19.2	16.4	10.9	5.5
MSMA	27.2	22.6	20.4	18.1	15.8	13.6	9.1	4.5
$Ti(OBu)_4$	40.0	50.0	55.0	60.0	65.0	70.0	80.0	90.0

[a] Note: All of the polymerization mixtures had the following fixed composition: (1) [MSMA]/([MMA]+[MSMA]) = 25 mol %, (2) [BPO]/([MMA]+[MSMA]) = 3.75 mol %, (3) $[H_2O]/[Ti(OBu)4] = 1$. (4) The amount of THF was adjusted so that the concentration of $Ti(OBu)_4$ was 1.71×10^{-4} mol/mL. (5) The reaction mixture was without acid catalyst.

Experimental Section

Materials. Methyl methacrylate (MMA, 99%, Lancaster), 3-(trimethoxysilyl)propyl methacrylate (MSMA, 98%, Aldrich), and tetrahydrofuran (THF, 99.9%, Acros) were purified by vacuum distillation. Titanium *n*-butoxide [$Ti(OBu)_4$, 99%, TCI] and benzoyl peroxide (BPO, 75%, Acros) were used as received.

Preparation of Hybrid Polymer Films. The compositions of the monomer mixtures for preparing polymer films are listed in Table 1. **T*X*** indicates the amount (wt %) of $Ti(OBu)_4$ in the reaction mixture. For example, **T90** means the weight ratio of the monomer $Ti(OBu)_4$ in the monomer mixture MMA + MSMA + $Ti(OBu)_4$ is 90%. The monomers, MMA and MSMA, and the initiator, BPO, were added into a reaction flask and were polymerized at 60 °C under nitrogen flow for 2 h. Then, a homogeneous THF solution of deionized water and $Ti(OBu)_4$ was added dropwise over 30 min into the reaction mixture with rigorous stirring to avoid local inhomogeneities. The reaction was allowed to proceed at 60 °C for another 2 h, and then the mixture was spin-coated at 3000 rpm on a silicon wafer for 40 s. The coated film was then cured at 80 °C for 1 h and 150 °C for another hour. The thicknesses of the prepared hybrid thin films **T40**–**T90** are in the range of 511–4595 Å, as shown in Table 2. The molar ratio of MSMA to MMA + MSMA was 0.25. The amount of deionized water was fixed at the mole ratio of $H_2O/Ti(OBu)_4 = 1$.

The amount of the polymerization solvent THF significantly affected the precipitation of inorganic particles or the gelation of reactants MSMA and $Ti(OBu)_4$ if sufficient solvent was not provided. It was found that the THF amount should keep the $Ti(OBu)_4$ concentration in the reaction mixture less than 1.80

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Table 2. Properties of the Prepared Materials

	T**0**[a]	T**10**[a]	T**20**[a]	T**25**[a]	T**33**[a]	**T40**	**T50**	**T55**	**T60**	**T65**	**T70**	**T80**	**T90**	**T100**[a]
T_d/°C	257	277	235	258	248	267	275	267	281	278	263	272	271	−
900 °C residue[b]	24.9	31.7	34.5	45.9	44.0	45.0	49.3	51.5	56.3	59.1	62.4	74.7	77.2	95.1
900 °C residue[c]	10.9	13.4	16.5	18.2	21.3	25.8	31.0	34.1	37.6	41.6	46.2	57.8	74.4	100.0
h[d] (Å)	5285	10043	2620	5010	3275	4595	2108	1894	1431	1199	947	630	511	604
R_a[e] (Å)	3.0	2.5	3.8	5.5	3.3	3.8	3.5	7.7	5.7	5.6	3.8	3.8	6.0	2.5
R_q[e] (Å)	3.8	3.2	4.7	7.0	4.3	4.9	4.4	9.7	7.2	7.0	4.8	4.8	8.1	3.2
$n_{632.8\text{nm}}$[f]	1.491	1.505	1.531	1.546	1.553	1.590	1.612	1.625	1.630	1.664	1.710	1.824	1.867	2.010
ν[g]	36.5	42.8	30.2	49.1	48.4	42.0	24.5	27.5	25.3	24.6	24.7	18.9	12.6	14.2

[a] Part of the data from a previous publication. [b] Experimental results from TGA. [c] Theoretical values based on the assumption that only inorganic moieties are present at 900 °C. [d] h = film thickness. [e] R_a and R_q are the average and root mean square roughness, respectively. [f] n = refractive index. [g] ν = Abbe number.

$\times 10^{-4}$ mol/mL to avoid the precipitation of TiO_2 particles or the gelation of the reactants. Hence, the concentration of Ti(OBu)$_4$ was kept at 1.71×10^{-4} mol/mL for the polymerization, as shown in Table 1.

Preparation of the Titania Film T100 and Hybrid Films T10−T33. The preparation of the titania thin film **T100** and the hybrid films **T10**−**T33** was based on the method reported in the literature[18,19] and our previous publication.[16]

Characterization. Samples of the polymer thin films were prepared by spin-coating the precursor solution onto a KBr pellet, followed by curing at different stages of temperature as described above. Then, infrared spectra of these samples were recorded at room temperature using a Jasco model FTIR 410 spectrophotometer. Thermal analysis, thermogravimetric analysis (TGA), and differential scanning calorimetry (DSC) were performed under nitrogen flow using a Dupont model 951 thermogravimetric analyzer and a DuPont model 910S differential scanning calorimeter at heating rates of 20 °C/min and 10 °C/min, respectively. The TGA and DSC samples were prepared by spin-coating the precursor solution onto a glass substrate, followed by curing at different stages of temperature as described above. An atomic force microscope (Digital Instrument, Inc., model DI 5000 AFM) was used to probe the surface morphology of the coated films. The microstructure of the prepared hybrid materials was further examined by field emission scanning electron microscopy (FE-SEM, Hitachi, model 4000). An n&k analyzer (model 1200, n&k Technology, Inc.) was used to measure the refractive index (n) and the extinction coefficient (k) of the prepared films in the wavelength range of 190−900 nm. The thickness (h) of the prepared film was also determined simultaneously. The Abbe number of the prepared hybrid polymer films was calculated according to the following equation: $\upsilon = (n_D - 1)/(n_F - n_C)$, where n_C, n_D, and n_F are the refractive indices of the polymer film at 656, 589, and 486 nm, respectively.

Results and Discussion

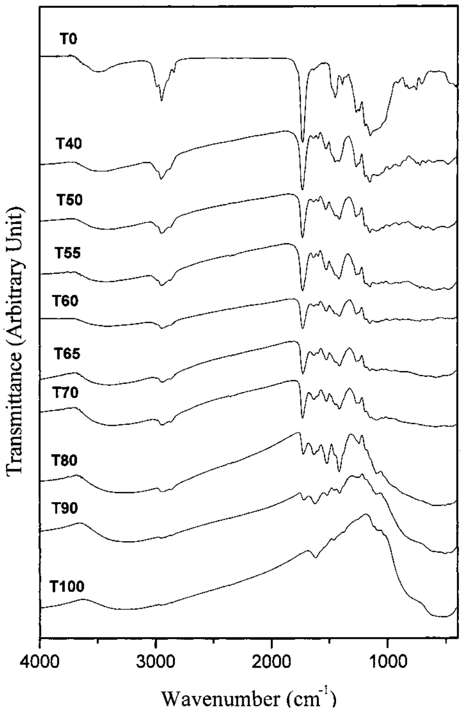

Figure 2. FTIR spectra of the poly(methyl methacrylate)−titania hybrid polymer films **T40**−**T90**, **T0**, and **T100**.

Figure 2 shows the FTIR spectra of the prepared poly(methyl methacrylate)−titania hybrid films **T0** and **T40**−**T100**. The assignments of the stretching vibration bands of the C═O and C−H bonds in the PMMA segment are at 1730 and 2950 cm^{-1}, respectively. The Ti−O−Ti and Ti−O−Si bands of the inorganic segment are observed from the broad absorption bands around 600 and 914 cm^{-1}, respectively.[20] These two bands gradually increase in intensity with increasing titania content, as compared to the intensity of the bands of the C═O band of the PMMA segment, which indicates that an increasing amount of titania is incorporated into the PMMA segment. The FTIR spectra of **T40**−**T100** exhibit an absorption band at 1630 cm^{-1}. However, this band did not appear in the spectrum of **T0**. Hence, the 1630 cm^{-1} band is probably not due to the C═C bond of the unreacted MMA residue after polymerization. It might arise from the absorption band of TiO_2 as this band appears in the spectrum of **T100** and its intensity increases with the titania content in the prepared hybrid materials. The Ti−OH band in the spectra of **T40**−**T100** is observed as a broad absorption in the range of 3400−3500 cm^{-1}. As discussed in the previous publication for **T0**−**T33**,[16] it might be due to the insufficient curing temperature as a high temperature greater than 400 °C is required for the synthesis of the pure TiO_2 materials. The position of the Ti−OH absorption band is below 3520 cm^{-1}. This is probably because of the hydrogen bonding of the Ti−OH residue group with the carbonyl group,[21] which makes the hybrid polymer film have good thermal stability and high optical transparence. Another possibility for the broad absorption band of the Ti−OH group is the aggregates of the hydroxyl groups. This is possible for the case of

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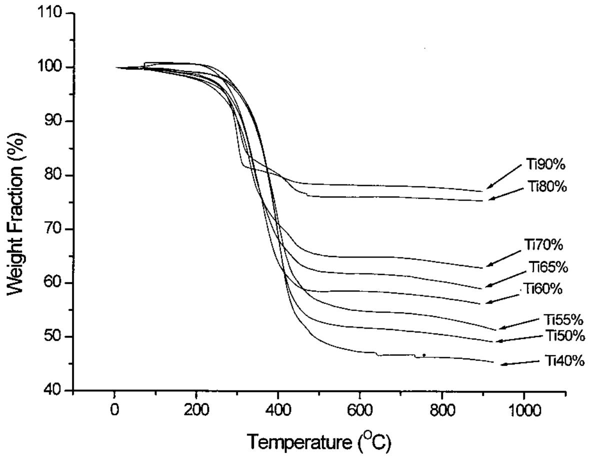

Figure 3. TGA curves of **T40**−**T90** at a heating rate of 20 °C/min under nitrogen flow.

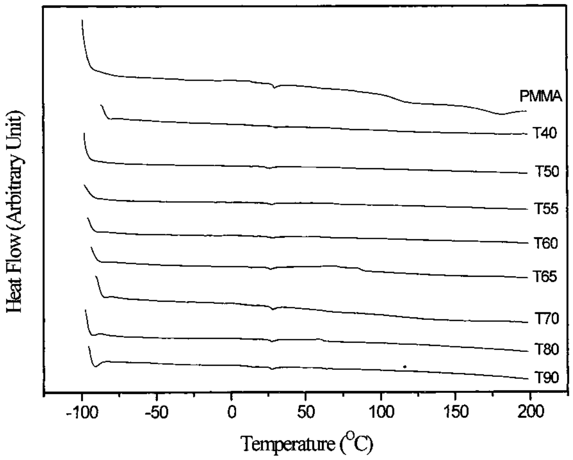

Figure 4. DSC curves of PMMA and **T40**− **T90** at a heating rate of 10 °C/min under nitrogen flow.

the hybrid materials with high titania contents such as **T80** and **T90**.

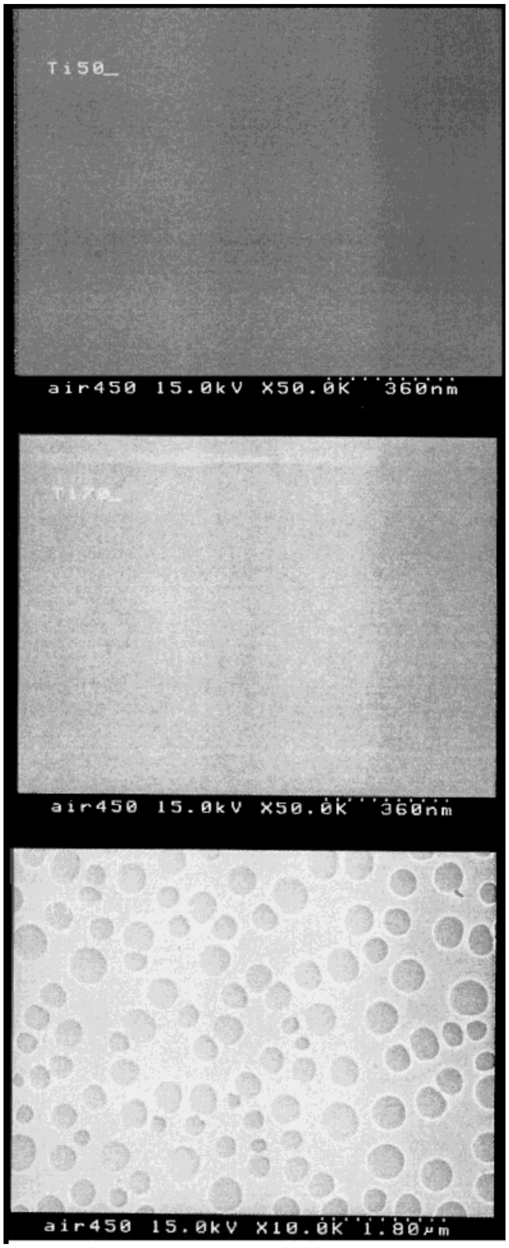

Figure 5. FE-SEM diagrams of (a) **T50**, (b) **T70**, and (c) **T90**.

Figure 3 illustrates the TGA curves of **T40**−**90** at a heating rate of 20 °C /min under nitrogen flow. The prepared hybrid polymer films have thermal decomposition temperatures (T_d) of 263−281 °C, as shown in Table 2. The T_d values are in a range similar to those of the previously prepared **T0**−**T33**, which is probably due to the decomposition of the PMMA segment. However, the char yields (polymer residues) of **T40**−**T90** at 900 °C increase with increasing titania content in the hybrid materials and are higher than those of the previously prepared **T0**−**T33**. They are in the range of 45−77.2%. This indicates that the $Ti(OBu)_4$ successfully incorporates into the network of the hybrid materials. The experimental char yield that is higher than the theoretical char yield shown in Table 2 is probably attributed to the organic moiety trapped in the inorganic matrix. The black color of the char after the TGA runs also provides evidence that the organic moiety has been trapped in the inorganic matrix. Figure 4 shows the DSC curves of PMMA and **T40**−**T90** at a heating rate of 10 °C/min under nitrogen flow. A small peak at around 25 °C in the DSC curves for all of the studied materials is due to water absorbed in the sample. The pure PMMA material has a T_g of around 113 °C. However, the hybrid materials **T40**−**T90** do not show any significant thermal transition peak that can be attributed to pure PMMA. This is probably because the inorganic moiety makes the polymer chain segment immobile, and thus, the glass transition temperature is probably close to the decomposition temperature. The DSC study also indicates good thermal stability of the prepared films and successful bonding of the organic and inorganic moieties in the hybrid films.

Figure 5 shows the FT-SEM images of the prepared hybrid materials (a) **T50**, (b) **T70**, and (c) **T90**. No significant TiO_2 domain size appears in the diagrams of **T50** and **T70**. According to the images in Figure 5a and 5b, the particle size of the TiO_2 in the hybrid materials is estimated to be probably smaller than 10 nm. Although the actual size of the TiO_2 domains cannot be precisely measured, the prepared hybrid materials **T50** and **T70** can be regarded as nanoscale materials from Figure 5a and b. A further study of the domain size by small-angle X-ray scattering can probably provide an accurate particle size of the TiO_2 in the hybrid materials. The FE-SEM images of the hybrid materials

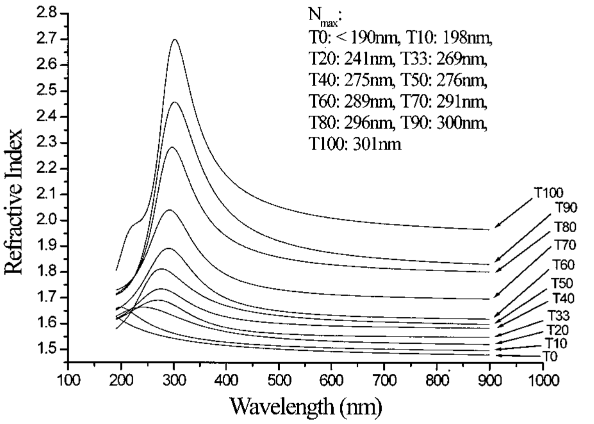

Figure 6. Variation of the refractive indices (n) of the hybrid films **T0** − **T100** in the wavelength range of 190−900 nm.

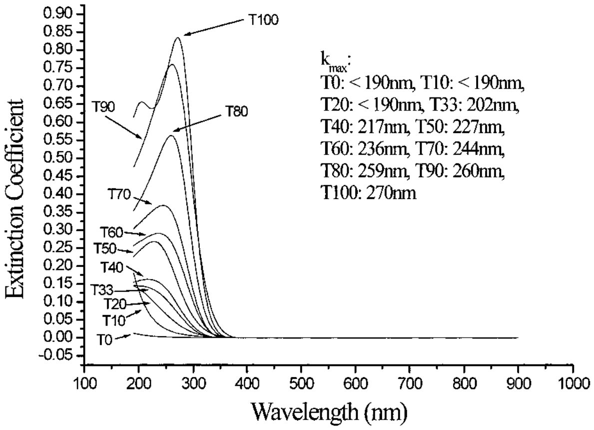

Figure 7. Variation of the extinction coefficients (k) of the hybrid films **T0**−**T100** in the wavelength range of 190−900 nm.

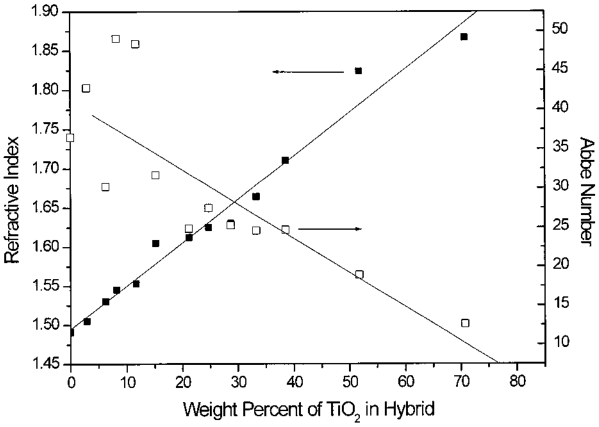

Figure 8. Variation of refractive index and Abbe number with the titania content (wt %) for the prepared films **T0**−**T100**.

T10−**T80** also show a domain size similar to that seen in Figure 5a and 5a. For the case of Figure 5c, TiO_2 particles within the size range of 100−400 nm are observed. This suggests that the hybrid material **T90** probably contained a long segment of TiO_2 before connecting with the PMMA segment. The size of the TiO_2 segment significantly affected its surface roughness and optical dispersion (e.g., Abbe number), which will be discussed in a later section. This result also indicates that the amount of $Ti(OBu)_4$ monomer for preparing the hybrid materials should be limited to obtain nanoscale materials. The average roughness (R_a) and root mean square roughness (R_q) of the prepared hybrid films **T10**−**T90** are shown in Table 2. The values of the roughness of the hybrid films compared with their film thickness are less than 0.5% for **T40**−**T70** and increase to more than 1.5% for **T90**. The surface roughness of the hybrid films **T40**−**T90** is higher than the that of the previously[16] prepared films **T10**−**T33**, which is probably due to the increasing titania size. This suggests good surface planarity of the prepared film. The result for **T90** indicates that the TiO_2 particle grows to a sufficient size, thus resulting in a surface roughness that is larger than those of the other hybrid materials. Another possibility is that the processing conditions might affect the surface roughness of the prepared hybrid materials.

Figure 6 shows the refractive index (n) distributions of **T0**−**T100** in the wavelength range of 190−900 nm. The refractive index of the prepared hybrid film at 633 nm increases from 1.505 for **T10** to 1.867 for **T90**, as shown in Table 2. The refractive indices of the prepared films **T40**−**T90** are higher than those of the previously prepared **T10**−**T33**. This suggests that increasing the incorporation of titania into the PMMA matrix results in an increment of the refractive index. The refractive index of **T100** is 2.01 at 633 nm, which supports the above argument. Figure 7 shows the extinction coefficients (k) of **T0**−**T100** in the wavelength range of 190−900 nm. The k values are almost zero for the studied films in the wavelength range of 400−900 nm, which indicates excellent optical transparency in the visible region. An interesting characteristic of Figure 7 is that the absorption maximum shifts from 198 nm for **T10** to 300 nm for **T90**. The absorption maximum of **T100** is similar to that of **T90**. The charge-transfer effect of the Ti−O−Ti segment increases with its size.[22,23] Hence, the absorption maximum of light shows a redshift with increasing titania content. As reported in the literature,[22] the shift of the peak maximum becomes significant when the TiO_2 particle size is less than 10 nm. As shown in Figure 5, the sizes of the TiO_2 segment of **T10**−**T80** are in the nanoscale region, unlike that of **T90**. Therefore, the redshift of the peak maximum shown in Figure 7 is due to the size effect of the titania segment. This also explains the absorption spectra of **T10**−**T33**, which could not be clearly interpreted in our previous publication.[16] The similarity of the peak maxima between Figures 6 and 7 also indicates that the refractive index maxima shown in Figure 6 is due to the absorption of the TiO_2 segment. Figure 8 illustrates the variations of the refractive index and Abbe number of the hybrid polymer films with the TiO_2 content. The refractive index increases from 1.505 for **T10** with a titania content of 2.9 wt % to 1.867 for **T90** with a titania content of 70.7 wt %. However, the Abbe number decreases from 42.8 for **T10** to 12.6 for **T90** for the hybrid materials, as shown in Table 2. The relationship between the refractive index and the titania content is the reverse of the trend for the Abbe number. This suggests that the optical properties of the prepared

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hybrid films can be tuned through their molecular compositions. The Abbe number suggests the optical dispersion of the prepared films. The absorption edges of the hybrid materials increase with the size of the TiO_2 segment, and therefore the Abbe number decreases. The refractive indices of **T40**−**T90** can be compared with those of the other high-refractive-index materials reported in the literature.[6,13] However, the low Abbe numbers of the prepared hybrid materials **T80** and **T90** might limit their applications in optical devices because of the strong wavelength dependence of the refractive index.

Conclusions

The high-refractive-index PMMA−titania hybrid thin films **T40**−**T90** were successfully prepared by the catalyst-free sol−gel process combined with spin-coating and a multistep baking process. The FTIR and DSC studies indicate the successful bonding of the PMMA and TiO_2 segments. The FE-SEM results suggest that the TiO_2 segment in the hybrid materials **T10**−**T80** is of a nanoscale size. The AFM study suggests that the prepared hybrid thin films have good planarization. The refractive indices of the prepared hybrid thin films increase with titania content, whereas the Abbe numbers show the reverse trend. This can be explained by the size of the titania segment in the hybrid materials. The refractive indices of the prepared hybrid material can be compared with those of other kinds of high-refractive-index hybrid materials reported in the literature. Excellent optical transparence in the visible region was observed for the prepared hybrid films. The shift of the absorption maximum of the prepared hybrid thin films can be correlated with the titania content. The prepared hybrid thin films can potentially be used as optical thin films.

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