Fabrication of anti-reflection coatings on plastics using the spraying layer-by-layer self-assembly technique

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Abstract — Anti-reflection (AR) coatings on plastic substrates have been extensively investigated with the development of large-area LCD and LED displays. A robust AR coating on plastics requires strong adhesion to the substrate, precise thickness and refractive index, and abrasion resistance. In this paper, abrasion-resistant AR coatings were fabricated on polycarbonate substrates using the layer-by-layer spraying deposition of poly(allylamine hydrochloride) (PAH) and silica nanoparticles. The adhesion between the substrates and coatings was enhanced by treating the polycarbonate surfaces with aminopropyltrimethoxylsilane (APTS). The porous low-refractive-index PAH/silica-nanoparticles multilayers were constructed by the layer-by-layer spraying of PAH and silica-nanoparticles aqueous solutions onto the functionalized substrates. The subsequent treatment of the porous coatings with tetrahydroxylsilane leads to stable abrasion-resistant AR coatings. The resultant AR coatings can reduce the reflection from 5 to 0.3%. The reported technique provides a cost-effective method for large-scale production of AR coatings on plastic substrates.

Keywords — *Antireflection plastics, spray, layer-by-layer self-assembly.*

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1 Introduction

Anti-reflection (AR) coatings have numerous applications in optical and display devices due to their capability of removing ghost images and enhancing the transmittance of light. For the application in display devices such as CRTs, PDPs, and LCDs, the AR coatings should be designed to achieve the highest transmittance in the visible region. The first important homogenous AR coating developed was the single-layer film with an optical thickness that is equal to one-quarter of the wavelength at which the coating is effective.[1] In order to obtain zero reflectance, the refractive indices of the substrate n_S medium n_m, and coating material n_C must satisfy the relation $n_C = (n_S n_m)^{1/2}$. It means that to achieve zero reflectance at the interface between air ($n_m = 1$) and a plastic substrate ($n_S = 1.5–1.6$), the refractive index of the coating material should be ~1.25.[2] However, the most commonly used AR material (MgF_2) has a refractive index of 1.38. If a dense MgF_2 layer is applied to a plastic surface, the reflectance of the air/plastics interface is only reduced from 4 to 1.5%. Two approaches have been utilized to overcome such limitations. One solution is to use multilayer coatings with various refractive indices. Such approach can achieve broadband anti-reflection properties, but the materials with the exact refractive indices required by the theoretical design are not always available.[3,4] The other approach is to produce layers with a controlled porosity in order to reduce the refractive index of the layer to the required value of 1.25. However, this method is limited by the fabrication process and the narrow anti-reflection region. On the other hand, Mother Nature has provided a perfect example for efficient broadband anti-reflection coatings. It was first discovered by Bernhard in 1967 that the reflection from the corneas of night-flying moths was close to zero for the purpose of camouflage. Such a high-antireflective property is attributed to a surface layer in which the refractive index varies gradually from unity to that of the bulk material. Without such a layer, the Fresnel reflection coefficient at an interface of two media is equal to $[(n_1 - n_2)/(n_1 + n_2)]^2$, where n_1 and n_2 are the refractive indices of the media. However, if there is a gradual change of index, the net reflectance can be regarded as the resultant of an infinite series of reflections at each incremental change in index. Because of this continuous variation, each reflection comes from a different depth from the surface and will have a different phase. If the transition takes place over an optical distance of $\lambda/2$, all phases are present, there will be destructive interference, and the reflectance will fall to zero.[5]

With the development of large-area LCD and LED displays, AR coatings on plastic substrates have been extensively investigated. A robust AR coating on plastics requires strong adhesion to the substrate, precise coating thickness and refractive index, and scratch resistance. Up to now, vacuum-coating processes such as chemical vapor deposition (CVD)[6,7] and plasma-ion-assisted deposition (PIAD)[8] are the major techniques for producing interference coatings that require a thickness precision in the range of 1 or 2 nm. However, a vacuum-coating process is not cost effective. Another approach to fabricate AR coatings uses wet-chemical coatings such as sol-gel coatings where the low-refrac-

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tive-index layer is generated by baking at a high temperature and burning out the organic particles which are in a suspension within a sol-gel coating.[9–11] Such an approach is only applicable to temperature-stable substrates instead of plastics.

On the other hand, the layer-by-layer (LBL) molecular-level adsorption of materials through different interactions is now a well-established methodology for creating conformal thin-film coatings with precisely tuned physical and chemical properties. This technique involves sequential adsorption of materials that can form intermolecular interactions. Intermolecular interactions including opposite electrostatic interactions,[12] hydrogen bonds,[13,14] and acid–base interactions[15] have been used in building layer-by-layer self-assembled multilayer systems. The monolayer growth of the film is a bottom-up approach and allows the precise control of film composition and dimension in nanoscale. Such technique provides a versatile platform for the assembly of materials and nanostructures of interest in making AR coatings.[16] Besides the conventional dipping procedure, the coatings can be fabricated by a spraying technique, which has the potential to meet the demand for techniques that create AR coatings on plastics in mass production.

In this paper, we report the fabrication of abrasion-resistant AR coatings on polycarbonate (PC) substrates using the aqueous-solution-based layer-by-layer assembly of poly(ally amine hydrochloride) (PAH, positively charged) and silica nanoparticles (negatively charged) combined with a sol-gel treatment. The low-refractive-index porous PAH/silica-nanoparticle coatings were fabricated by alternating sprays of PAH and silica-nanoparticle solutions onto the substrates. The scratching resistance was introduced by treating the PAH/silica nanoparticle porous layer with a sol-gel solution. The resultant AR coatings are able to reduce the reflection from 5 to 0.3%. Such a technique will provide a simple, cost-effective approach to build AR coatings on large area plastic substrates.

2 Experimental

2.1 Chemicals and materials

3-Amino(propyltrimethoxysilane) (APTS), nitric acid (HNO_3), ethanol (anhydrous, 98%), poly(allyamine hydrochloride), colloidal-silica 30-wt.% suspension in water, and tetraethylorthosilicate (TEOS, 98%) were purchased from Sigma-Aldrich (St. Louise, MO). The chromatographic spray bottles were purchased from Fisher Scientific (Pittsburgh, PA). Deionized (DI) water (18 MΩ) was obtained using a Milli-Q water system.

2.2 Polycarbonate surface functionalization with APTS

Polycarbonate substrates were immersed in a 5% (by weight) solution of 3-amino(propyl-trimethoxylsilane) (APTS) in isopropanol for 2 hours. The sample was then rinsed with isopropanol, followed by DI water.

2.3 Sol-gel preparation

A sol-gel stock solution was prepared by mixing 40 ml of tetraethylorthosilicate (TEOS) with 38.7 ml of ethanol and 4 ml of DI water. 3 ml of 0.1 M HNO_3 was added slowly to the solution. The solution was then magnetically stirred for 1 hour and was left to age for 24 hours. The hydrolysis of TEOS leads to the formation of tetrahydroxylsilane.

2.4 Spin coating of the sol-gel solution

The sol-gel solution was spin-coated on the substrates using a spin coater purchased from Laurel Technology Cooperation. Different spin rates (1000, 2500, and 2750 rpm) were used to control the film thickness.

2.5 Layer-by-layer (spraying) deposition

Ultrathin poly(allylamine hydrochloride) (PAH)/silica-nanoparticles nanoporous films were fabricated by an alternating deposition of PAH and silica nanoparticles by using a unique spraying technique. A 0.01 M solution of PAH with an adjusted pH of 6.8 and 0.1% (by weight) solution of silica nanoparticles with an adjusted pH of 6.8 were used to create the multilayer films. A constant flow of 120 psi (827.37 kPa) of nitrogen gas was used to spray the solutions with the chromatography spray bottles with a nozzle diameter of 1 mm. A layer of PAH was deposited on to the surface by spraying the PAH solution with two cycles of 3-sec spray/5-sec delay followed by another 3-sec spray and 10-sec delay. The substrate was then rinsed with DI water consisting of 2 cycles of 10-sec spray/5-sec delay. The silica nanoparticles were then applied following the same procedures as the PAH, and was then followed by another rinsing cycle. Such a procedure would generate a bi-layer of the AR coatings on the polycarbonate substrate.

2.6 Layer-by-layer (dipping) deposition

AR coatings were also fabricated via a dipping deposition to compare the AR properties with the AR properties of those fabricated *via* the spraying deposition. The polycarbonate substrate was first dipped into a cationic solution for 15 minutes followed, by one 2-minute and two 1-minute rinsing steps using deionized water. Then, the substrate was dipped into an anionic solution for 15 minutes, followed by the same rinsing steps.

2.7 Characterization

The coating thickness and refractive index was obtained using a J. A. Woollam Visible Variable-Angle Spectroscopic Ellipsometer (V-VASE) (0.2–1.1 μm). The transmittance of the samples was measured using a Varian 300 UV-Visible Spectrometer. The absorbance of the polycarbonate substrate was measured to be about 0.05% from 400 to 700 nm using a Varian 300 UV-Visible Spectrometer. The reflectance of the samples is calculated by substrate transmittance and absorbance from 100. The water contact angle of the samples was measured using a Rame'-Hart, Inc., Imaging System. The atomic-force microscope (AFM) images were obtained using a Dimension 3100.

3 Results and discussion

Polycarbonates (PCs) are tough, dimensionally stable, and transparent thermoplastics with extensive applications such as the automotive displays, lenses for eyeglasses, and non-automotive vehicle windows. Since a PC substrate has a high refractive index (1.58)[17] that leads to more than a 5% reflection on each side, all these optical surfaces require AR coatings to avoid unwanted reflections, improve the contrast of the displays, and increase the transmittance of light. AR coating on the optical glass is a well-established field, whereas the coating on the polymers is under extensive investigation. A robust AR coating on polycarbonate with high optical performance and mechanical properties requires strong adhesion to the substrate, precise coating thickness and refractive index, and abrasion resistance. In this study, various approaches including surface modification, layer-by-layer spraying deposition of PAH and silica nanoparticles, and sol-gel annealing have been applied to build robust AR coatings on polycarbonate substrates.

3.1 Modification of the PC substrate with APTS

The mechanical properties of the AR coatings on the polycarbonate substrates depend on the interfacial properties between coatings and substrates. Polycarbonates are polymers having functional groups linked together by carbonate groups, which are not able to provide strong adhesion with silica-based AR coatings. In order to generate silanol (Si–OH) groups on the polycarbonate surfaces, the substrates were immersed in a 5% 3-APTS solution for 2 hours, where the 3-APTS slightly penetrated through the PC surface and reacted with the carbonate group (Fig. 1).

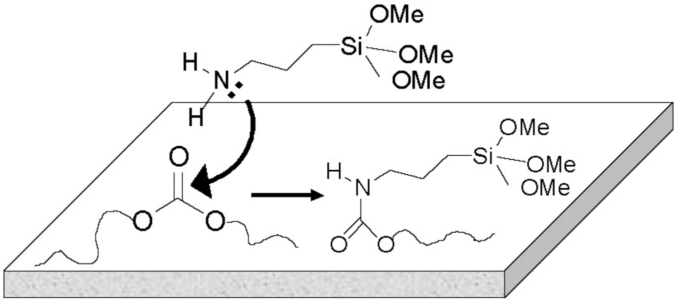

FIGURE 1 — Schematic illustration of the functionalization of PC with 3-APTS.

The chemical bonding mechanism of the 3-APTS and polycarbonate surface is based on the aminolysis of the carbonate groups. The selective chain scission of the polycarbonate enabled the trimethoxysilyl groups to react with carbonate groups to form urethane linkage.[18] When the samples were treated with water, the reacted and unreacted 3-APTS molecules were partially hydrolyzed and condensed on the surface as a thin layer of silanol groups. The chemical composition of a 3-APTS modified polycarbonate surface was examined by x-ray photoelectron spectroscopy (XPS) which clearly indicated the presence of silica (Si 2p^3) and nitrogen (N 1S) peaks [Fig. 2(b)]. This XPS survey spectrum of was compared to that of a bare polycarbonate substrate [Fig. 2(a)] and a 3-APTS treated glass substrate, which served as a free-amine reference sample [Fig. 2(c)]. The

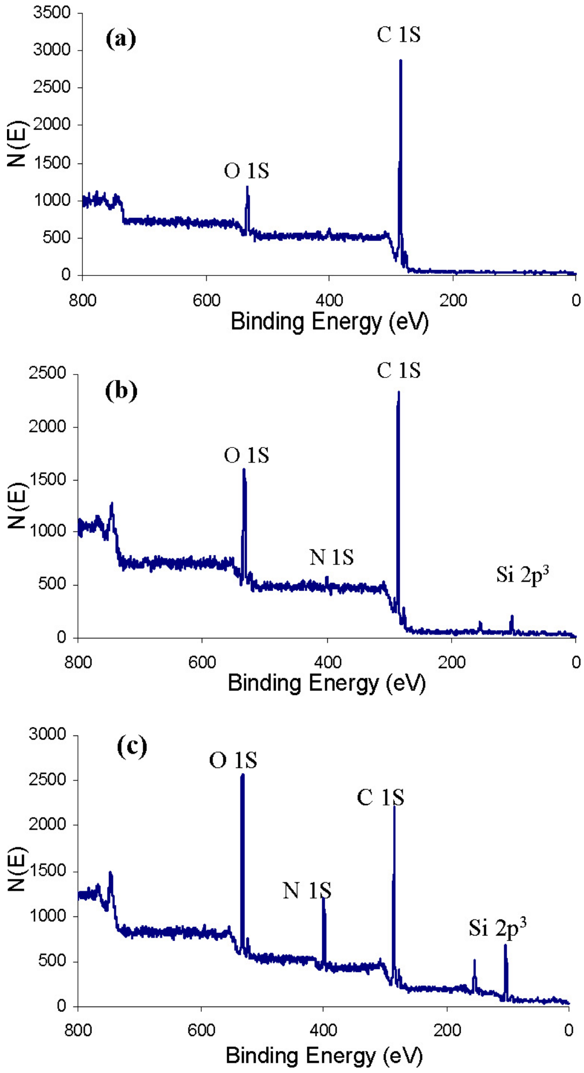

FIGURE 2 — XPS spectra of (a) a bare PC substrate, (b) an APTS-modified PC substrate, and (c) an APTS-modified glass sample.

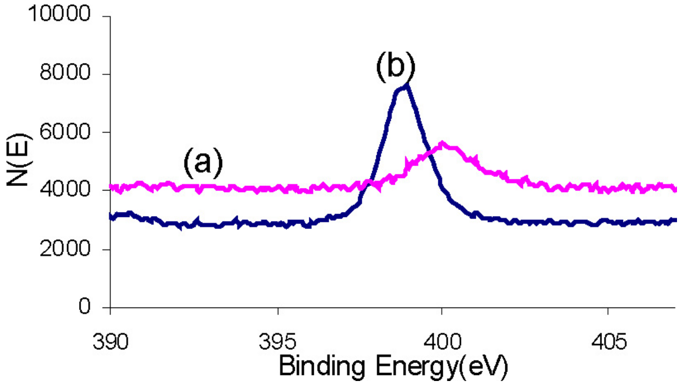

FIGURE 3 — Binding energies of the N 1S electrons in (a) urethane linkage of APTS-modified PC surface and (b) free amine of APTS-modified glass surface as obtained from high-resolution XPS analysis.

XPS data of the samples would give the chemical information of the free-amine nitrogen and the urethane nitrogen and ensure the aminolysis reaction on the PC surface. The bare polycarbonate substrate had only carbon (C 1S) and oxygen (O 1S) peaks [Fig. 2(a)] which were enhanced in the XPS spectra of the APTS-modified PC sample [Fig. 2(b)]. The enhancement of the carbon and oxygen peak was due to the methoxy groups and the propyl group of the APTS molecules attached to the PC surface. The free-amine nitrogen of the APTS-modified glass surface and the nitrogen of the urethane linkage on the APTS-modified PC substrate could be clearly distinguished by comparing the binding energies of the N 1S electrons obtained from the high-resolution XPS spectrum of the respective samples (Fig. 3). The high-resolution XPS data showed that the binding energy of the N 1S electron for the 3-APTS modified PC samples was at 400.0 eV [Fig. 3(a)], while the binding energy of the N 1S electron for the free-amine reference (glass substrate) was at 398.9 eV [Fig. 3(b)]. Therefore, the XPS nitrogen peak of the APTS-modified PC indicates the formation the urethane groups instead of amine groups.

Additionally, the thin APTS layer on the PC substrate increased the hydrophilicity of the surface, which was confirmed by the contact-angle measurement as in shown Fig. 4. After the surface modification, the water contact angle changes from 90° to 40°. The increased wettability of the PC surface facilitated the subsequent steps of spray coating and the sol-gel deposition.

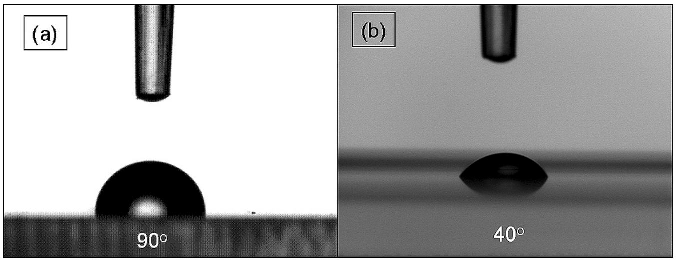

FIGURE 4 — (a) A bare PC sample showing contact angle of 90° and (b) an APTS-modified PC sample showing contact angle of 40°.

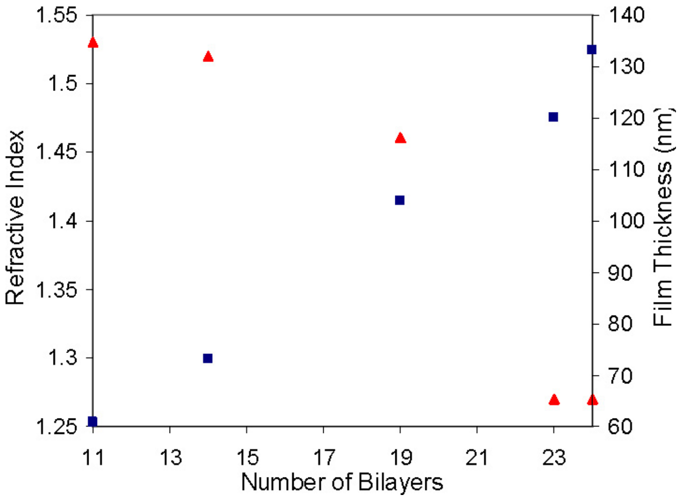

FIGURE 5 — The film thickness and refractive-index changes with a number of bilayers deposited (■ symbolizes film thickness and ▲ symbolizes refractive index.)

3.2 Layer-by-layer deposition of PAH/silica-nanoparticle AR coatings on APTS-modified polycarbonate substrates

The AR coating was fabricated on the APTS-modified PC surface by the alternating layer-by-layer spraying deposition of PAH and silica-nanoparticle solutions with intermediate rinsing cycles. To achieve the lowest reflection, the refractive index of the AR coatings should be 1.26, the square root of the refractive index of the PC substrate. Different deposition parameters including the pH of the solutions, the number of layers, and rinsing time were optimized to achieve the highest transmittance with the least number of spraying cycles. In our studies, the refractive index of the coating decreases with the number of PAH/silica-nanoparticles bilayer deposited, which is consistent with the results reported by Rubner and co-workers[15] (Fig. 5). Figure 6 shows the UV–visible transmittance spectra of a sample with 11 bi-layers to 24 bi-layers of PAH/silica nanoparticles depos-

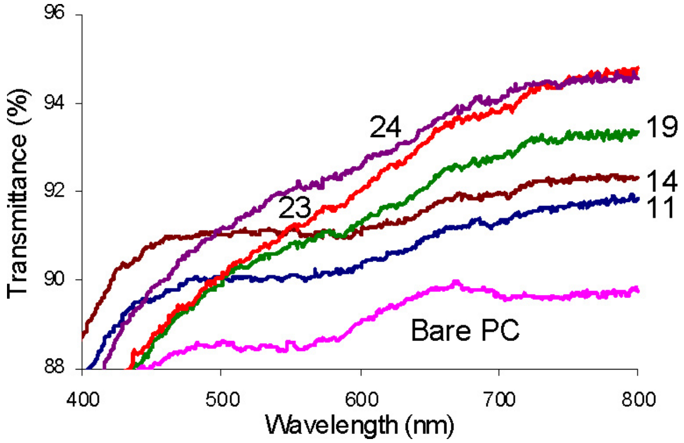

FIGURE 6 — UV–visible transmittance spectra of the AR-coated PC substrate with a different number of bilayers as compared to a bare PC substrate.

ited on one side of a PC substrate. In Fig. 6, the transmittance of the coating increases with the film thickness due to the decrease of the refractive index. The highest transmittance (94.6% at 700 nm) is obtained using an AR coating containing 24 bi-layers of PAH/silica nanoparticles with a refractive index of 1.27. Since one side of the polycarbonate substrate is coated with the AR coating while both sides contribute to the reflection. The reflectance of the coated sample includes 5% reflectance of the uncoated side and the reduced reflectance of the coated side. Therefore, the reflectance of the coated side can be calculated by subtracting 5% (reflectance of uncoated side), 94.6% (transmittance at 700 nm), and 0.05% (absorbance) from 100%, which leads to 0.35%. If the transmittance at 550 nm (93%) is considered, the reflectance is reduced from 5 to 1.95%.

The adhesion between the AR coating and the APTS-modified substrate was examined using a Scotch Tape test where a tape is pressed on the AR coating and peeled off at 45°. The AR coatings on the APTS-modified PC carbonate were undisturbed while the AR coating on the bare PC substrate was completely removed with the tape. This result suggests that APTS-modified PC surfaces provide sufficient functional groups to form strong bonding with silica-based coatings. APTS-modified PC substrates were also used in the subsequent studies of sol-gel-modified AR coatings.

3.3 Sol-gel coating on APTS-modified PC substrate

It is also observed in Fig. 6 that a steep drop in transmittance occurs for the coating with 24 bi-layers of PAH/silica nanoparticles at short wavelength, and the reflection is only reduced from 5 to 2% at 550 nm. Such a narrow AR region is probably due to the large difference between the refractive index of the polycarbonate substrate (1.58) and that of the AR coating (1.27). Reducing the refractive-index difference and building an index gradient has been demonstrated by nature to be an efficient approach to generate high-quality AR coatings. A broadband AR coating with high transmittance in the visible light region has been fabricated by using two layers of coatings with different refractive indices.[19] Therefore, it is expected that the AR properties at short wavelength will be improved by incorporating one layer of coatings with an refractive index between 1.58 and 1.27 between the APTS-modified PC substrate and the porous AR coating. A sol-gel solution (1.88% by weight) was spin-coated on one side of the APTS-modified PC substrate with a spin rate of 2750 rpm. After aging at room temperature overnight, the sol-gel coating formed a uniform strong silica coating on the APTS-modified PC through the formation of siloxane (Si–O–Si) bonds. The sol-gel layer had a refractive index of 1.52 and thickness of 61 nm. As shown in Fig. 7, such a thin silica layer has AR properties because it has a lower refractive index than the PC substrate.

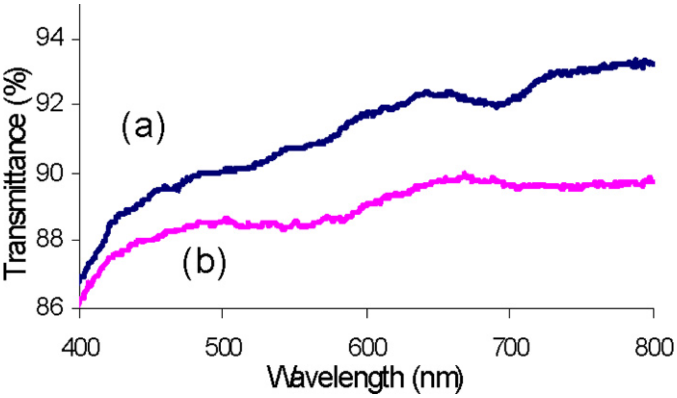

FIGURE 7 — UV–visible transmittance spectra of (a) a sol-gel-modified PC substrate and (b) a bare PC substrate.

Besides improving the AR properties, the sol-gel layer enhanced the attaching of PAH/silica nanoparticles onto the substrate in the layer-by-layer spraying deposition due to the high density of silanol groups on the surface. It was found that the number of bi-layers on the sol-gel-modified surface to achieve optimum transmittance was much less than the number of bi-layers on the APTS-modified substrates. Only 10 bi-layers of PAH/silica-nanoparticle coating on one side of the sol-gel-modified PC substrate are able to produce AR coatings with 94.5% transmittance. In contrast, 24 bi-layers of PAH/silica nanoparticles are required on the APTS-modified substrate to achieve the comparable result (Fig. 8). The AR coatings on the sol-gel-modified surfaces had a broad-band transmittance of 93% over a range of 600–800 nm. Additionally, the AR coating on the sol-gel-modified PC [Fig. 8(b)] has 1% higher transmittance at 500 nm than the AR coating on the APTS-modified PC [Fig. 8(c)].

Compared with a conventional dipping method, the spraying technique has the potential for mass production. Therefore, it is important to compare the AR qualities of AR coatings fabricated using dipping and spraying deposition. As shown in Fig. 8(a), the sol-gel-modified PC substrate dipping coated with 14 bi-layers of PAH/silica nanoparticles has a similar transmittance in the range of 600–800 nm but higher transmittance at shorter wavelengths compared with

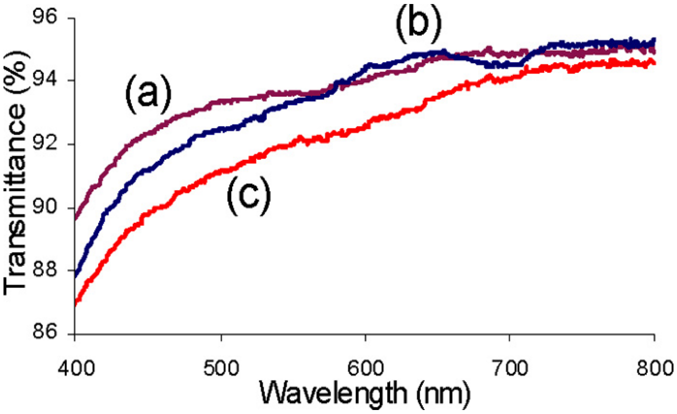

FIGURE 8 — UV–visible transmittance spectra of (a) 14 bi-layers of AR coating on one side of a sol-gel-modified PC substrate by dip-coating, (b) 10 bi-layers of AR coating on one side of a sol-gel modified PC substrate by spraying deposition, and (c) 24 bi-layers of AR coating on one side of the APTS-modified PC substrate by spray deposition.

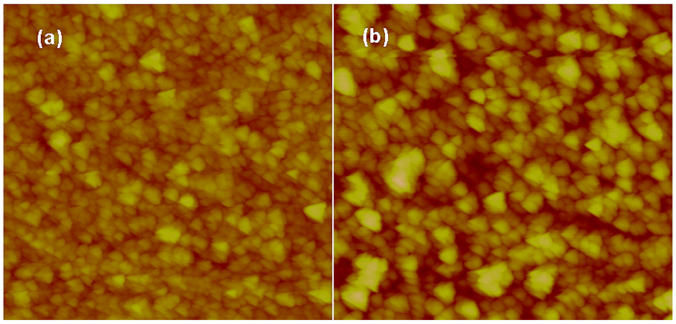

FIGURE 9 — AFM images of (a) 14 bi-layers of AR coating on one side of sol-gel-modified PC substrate by dip-coating and (b) 10 bi-layers of AR coating on one side of the sol-gel-modified PC substrate by spray coating. The scale of the images is 5 µm.

the sol-gel-modified PC substrate spraying coated with five bi-layers of PAH/silica nanoparticles [Fig. 8(b)]. Such dissimilarity is attributed to the different packing density of the silica nanoparticles in dipping and spraying deposition. The surface morphology of the spraying-coated and dipping-coated AR PC samples were analyzed by an atomic-force microscope (AFM). As shown in Fig. 9, silica nanoparticles deposited via a spraying approach form looser structures than those deposited via a dipping approach. The loose structure leads to larger thickness and smaller refractive index for each bi-layer of PAH/silica nanoparticles. Therefore, the less number of bilayers of PAH/silica nanoparticles is required to achieve comparable AR properties using the spraying deposition.

3.4 Fabrication of abrasion-resistant AR coatings

As discussed previously, the spraying layer-by-layer deposition of PAH/silica nanoparticles provides a cost-effective approach to build AR coatings in PC substrates. However, the AR coatings are very fragile due to the weak bonds (electrostatic bonds) between PAH and silica nanoparticles. The AR coatings can be easily damaged by wiping them with Kimwipes. Therefore, it is essential to toughen the AR coatings for their practical applications. Rubner and co-workers have made strong AR coatings from polyelectrolyte/silica-nanoparticle systems through a hydrothermal treatment where the hot steam (124–134°C) catalyzed the fusing of neighboring silica nanoparticles.[20] In our studies, the mechanical performance of the AR coating was improved by treating the porous AR coatings with a sol-gel solution (1% by weight). After the sample was heated at 100°C overnight, the silica nanoparticles were fused through the reaction with tetrahydroxylsilane in the sol-gel solutions, leading to a uniform hard AR coating. The abrasion-resistant test was evaluated by rubbing the coatings with Kimwipes. No scratches formed on sol-gel-treated coatings, suggesting the abrasion resistant properties. The UV–visible transmittance of the antireflective PC samples after the sol-gel annealing treatment is shown in Fig. 10, which indicates that the sol-gel treatment did not affect the antireflection properties of the AR coating. In Fig. 11, the PC with AR coatings (left slide) shows much lower reflection than the bare PC (right slide). The AR coating not only greatly enhanced the transmittance, but also was abrasion resistant when tested by rubbing with Kimwipes.

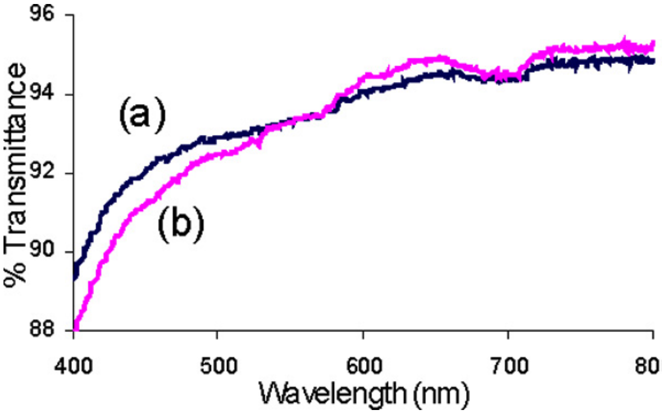

FIGURE 10 — Top panel: UV–visible transmittance spectra of (a) an AR coating on a PC substrate with the sol-gel treatment and (b) an AR coating on a PC substrate without the sol-gel treatment.

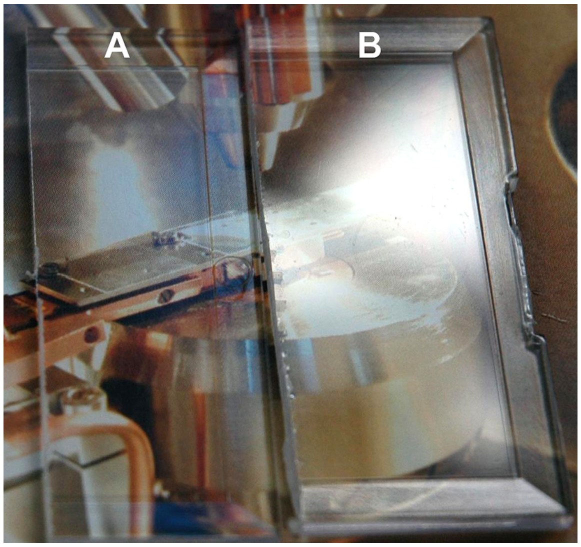

FIGURE 11 — A photograph of PC substrates (a) with and (b) without AR coatings.

4 Conclusion

In summary, abrasion-resistant AR coatings on polycarbonate substrate were fabricated using spraying layer-by-layer deposition of PAH/silica nanoparticles. The mechanical properties of the coatings were improved by the functionalization of PC surface and sol-gel treatments. The resultant abrasion-resistant AR coatings are able to reduce the reflection from 5 to 0.3%. The reported technique provides a cost effective and versatile approach to fabricate conformal AR coatings on plastics with the potential of mass production.

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