



Preparation of hydrophobic and abrasion-resistant silica antireflective coatings by using a cationic surfactant to regulate surface morphologies

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Abstract

Antireflective (AR) coatings were prepared in the process of the acid catalyzed sol–gel process by using tetraethylorthosilicate (TEOS) as silica precursor and cetyltrimethyl ammonium bromide (CTAB) as template. Acid-catalyzed silica AR coatings exhibited extremely strong adhesive forces with glass substrates and excellent abrasion-resistant property. The experimental results revealed that the regulation of the template concentrations in silica sols could not only improve the transmittance of coatings but also alter the surface morphology and roughness. To further improve hydrophobicity and minimize the conflict between roughness and transmittance, the target coating was designed as a bilayer coating, composed of a porous bottom layer acting as AR coating and a rough top layer providing applicable roughness. Trimethylchlorosilane (TMCS) treatment was finally used to obtain the hydrophobicity of the AR coatings. The results indicated that, the antireflection, hydrophobicity and abrasion-resistant property were successfully integrated in the bilayer AR coating, which showed 96.6% transmittance, 130° water contact angle and 5 H pencil hardness. Meanwhile, the multiple performances are very promising in the long practical application of solar collector covers.

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1. Introduction

In the trough concentrating solar power (CSP) systems, the efficiency of producing electricity greatly depends on the intensity of the incident sunlight. A basal drawback of any transparent cover materials used to create the vacuum for thermal insulation is the main reflection of the incident sunlight. The application of antireflective (AR) coatings on solar collector covers is a popular method to reduce the reflection (Nostell et al., 1999; Hensch et al., 2010; Vicente et al., 2009). In consideration of practical application, AR coatings are also anticipated to obtain well abrasion-resistant property and certain

hydrophobic self-cleaning capacity, a guarantee for CSP systems to perform efficiently for long in wild and harsh environment.

For the advantage of easy operation and low cost, the normal sol–gel process has become a widely used method to produce silica AR coatings (Xiu et al., 2009; Hensch and Deubener, 2012; Manca et al., 2009; Nostell et al., 1999; Huang et al., 2009; Bautista and Morales, 2003; Li et al., 2013). Especially in the application of covering both sides of those non-flat evacuated solar thermal glass tubes, the sol–gel method occupies a unique position. The normal silica sol, synthesized by the typical base-catalyzed Stöber method, is chiefly composed of silica particles (Stöber et al., 1968; Zhang et al., 2010). Since only physical forces, instead of chemical bonds, exist among individual silica particles and between such particles and the glass substrate surface, the

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coatings will be irreversibly damaged even by gentle external forces (Chi et al., 2011a,b). The AR coatings used for solar thermal collectors need not only higher transmittance but also outstanding abrasion-resistance to the harsh and open conditions and external cleaning. Yet, by using another acid-catalyzed process, the formation of the polysilicates occurs by cluster–cluster aggregation, resulting in highly branched polymers (Chi et al., 2011a,b; Ye et al., 2011). In the process of coating, as the solvent evaporates, the branches create more hydroxyl (–OH) and oxyethyl (–OEt) groups, which are known to promote the formation of chemical bonds and structure densification in the course of calcination (Mehner et al., 2010). As a result, the mechanical property of acid-catalyzed coating is dramatically improved by these internal cross-linked chemical bonds (Ye et al., 2011; Mehner et al., 2010), in other words, the acid catalyzed coatings possess much better abrasion-resistant property. However, the coatings prepared from acid-catalyzed sols present extremely dense structure, which results in a high refractive index and, consequently, low transmittance. Because the functionality of the antireflection silica coatings is based on its nanoporosity (Helsch et al., 2010), creating more pores inside the dense coatings can achieve the aim of reducing the refractive index and increasing the transmittance.

On the other hand, the stability of AR films in extensive applications is also a vital aspect that should be taken into account. Normally, the porous coatings are usually rich in residual silanol groups (Si–OH). In the actual application, the superhydrophilic silanol groups are very active and prone to induce the adsorption of water vapor and contaminants in humid or wild surroundings, leading to the deterioration of the AR properties (Vicente et al., 2009). Furthermore, the durability and self-cleaning capacity of the coatings tend to increase with the improvement of hydrophobicity of the coating surface. Thus, to guarantee its practicality and endurance, the surface of AR films should possess such hydrophobic property. As is well known, the hydrophobic abilities are determined by two basic factors: low surface energy and rough surface structure of the surface. Post-grafting organosiloxanes have been proved to eliminate the silanol groups and to decrease the surface energy (Xu et al., 2005; Akamatsua et al., 2001). Nevertheless, the smooth solid surface which is even modified by $-\text{CF}_3$ groups with the lowest surface energy 6.7 mJ/m^2 , gives a water contact angle (WCA) of only 120° , still not a high level (Nishino and Meguro, 1999). On the other hand, elevating surface roughness can promote the increase of hydrophobicity, and WCA of more than 150° can be achieved (Nakajima et al., 2000). However, in the case of preparing AR coatings, the increase in surface roughness would be problematic as the transmittance of the films might be reduced due to the light scattering caused by rough structure (Vicente et al., 2011; Cho et al., 2010). Therefore, a balance between roughness and transmittance should be identified from the perspective of application.

In summary, most researches were engaged in the regulation of just one or two aspects in AR effects, hydrophobicity and abrasion-resistant property. In other words, there have been few reports in the study of all of these properties existing simultaneously. In this study, silica AR coatings were prepared by the acid catalyzed sol–gel process. Acid catalyzed silica coatings exhibited excellent abrasion-resistant property. The transmittances and surface morphologies of coatings were regulated by altering the concentrations of template in silica sols. After coating, TMCS treatment for AR coating surface was further used to obtain hydrophobicity so as to improve the practicality and durability of AR coatings.

2. Experimental

In this study, a dip-coating of acid catalyzed sol–gel templating method was used to prepare the porous silica coatings. In the process, tetraethylorthosilicate (TEOS) was used as silica precursor, concentrated hydrochloric acid as catalyst, absolute alcohol as solvent and cationic surfactant, hexadecyl trimethyl ammonium bromide (CTAB) as template. All the reagents were analytical grade and purchased from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China).

2.1. Preparation of acid-catalyzed templating silica sols

Concentrated hydrochloric acid (37% HCl mass fraction) and deionized water were first added into the alcohol. This mixture being stirred for a while, different amounts of CTAB were respectively dissolved in the previous mixture, which served as parallel experiments. Then, TEOS was dropwise added to the mixtures, which was stirred for 6 h at room temperature. Two series of sols with different silica concentrations to alcohol were synthesized, marked as sol-A and sol-B, respectively. As for sol-A, the molar ratio of TEOS:H₂O:EtOH:HCl was 1:4:25:0.03 and the mass concentration of CTAB in each sol-A was 0 g/L, 5 g/L, 10 g/L, 20 g/L, 25 g/L, 30 g/L, 40 g/L, respectively. In contrast, the molar ratio of raw material for sol-B was 1:4:100:0.03, successively, while the mass concentrations of CTAB were 25 g/L, 45 g/L, 55 g/L, 65 g/L, 75 g/L, respectively. All the resultant sols were aged in sealed beaker at thermostatic 20°C for 3 days before coating.

2.2. Cleaning procedure for the substrates

The procedure of cleaning up the substrates was operated as follows: the 3 mm-thick borosilicate glass substrates ($100 \times 25 \text{ mm}^2$) were successively treated by ultrasonic in basic solution and acid solution for an hour. The mixed basic and acid washing solution had the volume ratio of H₂O:H₂O₂:NH₃·H₂O = 5:1:1 and H₂O:H₂O₂:HCl = 5:1:1, respectively. Then, the substrates were completely cleaned up by deionized water and alcohol. Well cleaned substrates were dried off for use.

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