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A novel wet coating method using small amounts of solution on large flat substrates



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ABSTRACT

Coating on large surfaces is a critical issue in both academic studies and industrial production. This work proposes a novel method of coating a large flat substrate $(50 \times 100 \, \mathrm{cm^2})$ via a wet chemical process using a very small amount $(20 \, \mathrm{ml})$ of coating solution. The sol material consisted of surface-modified silicon dioxide (SiO_2) nanoparticles $(10-30 \, \mathrm{nm})$, which have the optimal antireflective (AR) function in the visible spectral range for thin films with a thickness ranging from 110 to 120 nm.

Ellipsometry results demonstrate a homogeneous thickness of the AR coating on glass (109.4 ± 2.7 nm). A deviation of less than 3% over a large coated surface was observed. Crack-free coatings with homogeneous morphology on the surface of the coatings were observed using scanning electron microscopy. The AR effect was confirmed with UV-vis measurements, with an average transmittance of 91.1% and 94.7%, respectively, in visible wavelengths for the one-sided and double-sided AR coatings (in comparison to 88% for uncoated glass).

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1. Introduction

A variety of coating techniques have been developed for deposition of thin films on substrates via the following methods: wet chemical processes, spin coating, spray coating, roll-to-roll coating, and dip coating [1–4]. Each technique has different advantages and disadvantages. For example, spin coating is useful for small (<30 cm in diameter), flat, and symmetrical substrates [5]. Spray coating is appropriate for arbitrary substrate shapes [6], but the thickness can be very inhomogeneous, which precludes coating thicknesses below 100 nm. Roll-to-roll coating is another method for large area coating, but it can only be used on flexible substrates, such as foils [7].

Among these various methods, dip coating is an alternative for deposition of thin films on a large substrate. Due to the notable disadvantage of the dip coating method, a large amount of sol must be used in comparison to other coating techniques [8]. Dip coating has attracted more attention than other methods because of its simplicity of use and its affordability for both academic research and industrial production; it also allows for the fabrication of homogenous coatings on different substrates [9]. In addition to being able to systematically control the thickness, this type of coating could

However, the biggest disadvantage of the conventional dip coating method is that it needs a large volume of solution when coating a big substrate. Therefore, it could result in a large amount of solution waste, which is unacceptable for large scale industrial applications especially when the solution is expensive or flammable.

We fabricated a novel coating device, which we call the "wiping-coating" method. This method only needs a few milliliters of solution for deposition on a large substrate [15]. This study aimed to apply this new coating method to coat large and flat substrates with a small amount of solvent. By overcoming the disadvantage of the dip coating process, our method could be applied to the fabrication of coatings on large and flat substrates, such as glass and flexible materials (PET, PEC, PMMA), for industrial application with a minimum amount of solution. Our results demonstrate that the proposed method leads to homogeneous coatings that enhanced the transmission to more than 94% for double-sided antireflective (AR) coated glasses.

2. Experimental section

Prior to the deposition, glass substrates ($50 \text{ cm} \times 100 \text{ cm}$ with a thickness of 3 mm) were washed and cleaned using ethanol, and then dried with nitrogen gas. Silicon dioxide (SiO_2) nanoparticles

be applied to all types of materials, including nanocomposites [10] and polymers [11], as well as a wide variety of dispersed materials [12–14].

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Fig. 1. The proposed wiping-coating device in order to coat large area substrates: (a) aluminum plate with holes connected to the vacuum pumps; (b) polycarbonate plate; (c) sol tank with adjustable speed and adjustable distance from the substrate; (d) processor controllers.

were dispersed in ethanol in order to coat the AR layer on the glass substrates. The substrates were coated using different speeds and subsequently annealed under the air ambient. The oven was heated with the rate of 5 K/min and then the temperature was maintained at 500 °C for 1 h.

The surface of the thin films was observed by scanning electron microscopy (SEM) (JSM-7500F; JEOL). Optical transmission of the thin films was measured with UV-vis spectrophotometer (Cary 5000; Varian). A variable angle spectroscopic ellipsometer (M-2000DI; J.A. Woollam) was used to measure the thickness of the films. The scratch resistance of the annealed thin films was analyzed with a pencil hardness test (Staedtler pencils with a hardness ranging from 8B to 6H) according to ASTM D3363 standards.

3. Results and discussion

3.1. Technical approach

The coating machine consists of a vertical frame $(2.2~m\times1.6~m)$ made of rigid aluminium to avoid vibration. The main part of the coating machine is the linear unit with an adjustable speed ranging from 0.05~mm/min to 600~mm/min.

The substrate holder consists of a large aluminum plate with orderly mounted holes that are connected to the vacuum pumps.

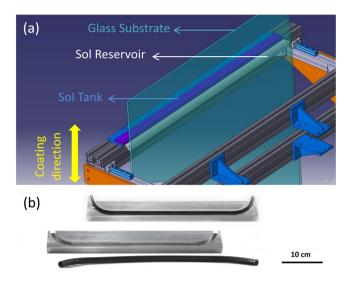
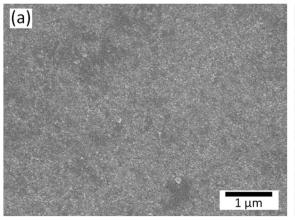


Fig. 2. (a) Schematic diagram of the coating device, (b) Construction of the sol tank with mounted rubber as the sol reservoir.

The glass substrate was attached by vacuum to the plate and it was fixed during the coating. To prevent the temperature inhomogeneity of the glass substrate, a polycarbonate sheet was mounted between the metal plate and the glass substrate to ensure thermal isolation (Fig. 1).

A schematic drawing of the coating system is shown in Fig. 2a. The aluminum tank ($5 \times 50 \times 5 \text{ cm}^3$) could be pressed softly onto the glass substrate using a mechanical construction (adjustable μm range). A rubber tube was mounted on one side of the tank, attached using a film tape with durable abrasion resistance in order to prevent abrasion between the glass substrate and rubber tube (Fig. 2b). The substrate was fixed, and the sol was uniformly poured inside the coating tank with a syringe mounted on a movable stage.

The pressure between the rubber tube and the glass substrate should be as tight as possible so that the sol cannot seep between the rubber tube and the substrate. The sol reservoir moves from the top of the substrate downward, using the processor controllers. By changing the coating speed, it is possible to vary the thickness of the coating. Generally, higher speeds lead to thicker layers. However, a higher speed could have a negative effect on the quality of the coating. Therefore, the speed of the coating should be optimized for each sol depending on the desired thickness, and the viscosity, concentration, and other properties of the sol. In this study, glass substrates were coated using the prepared SiO₂ sol at differ-



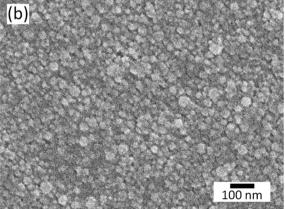


Fig. 3. Plan-view SEM images of the SiO₂ thin films on glass substrate: (a) morphology of the homogeneous coating obtained by lower magnification micro-graph and (b) the particle sizes less than 30 nm observed by higher magnification image.

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