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High performance single layer nano-porous antireflection coatings on glass by sol–gel process for solar energy applications

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ABSTRACT

Antireflection coatings have received a great importance due to their ability to enhance the efficiency of the solar cells and solar selective coatings by minimizing the reflections of the incident light from the front surface. In this study, a silica (SiO_2) sol, prepared using sol–gel process, was deposited on cleaned glass substrates by dip coating method and subjected to heat treatment at 400 °C. The thickness and porosity of the coating were optimized to achieve high transmittance. The thickness was optimized by varying the lifting speed of the substrate from the sol. The porosity was induced in the coating by using polymeric additives and through heat treatments. The optimized single layer SiO_2 coating on cleaned glass substrate exhibited a maximum transmittance of 97.5% at λ=500 nm wavelength. The hybrid sol was found to give reproducible coatings up to a period of 30 days when stored at 16 °C. The present process provides a simple and cost effective method for the preparation of antireflection coatings, which have huge potential to enhance the efficiency of solar cells, receiver tubes and other solar devices.
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1. Introduction

Antireflection (AR) coatings have been widely used to increase the solar radiation absorption in solar collectors to reduce front surface reflection of selective absorbers [1] as well as collector glass covers of solar selective coatings [2]. Silicon solar cells, with the band gap of 1.1 eV, convert light into electrical energy with a spectral response range of 380–900 nm. At present, the visible light transmittance of low iron glass, commonly used for the photovoltaic cells, is only ~92% [3,4]. The market prospects of antireflection coatings that can decrease light reflections from the surface of solar devices (both photovoltaic and photothermal) to increase efficiency of solar power generation are promising. Antireflection coatings reduce the reflection considerably by improving the quality of optical lens systems [5]. Many applications require an increase in the transmittance, or a reduction in the front surface reflection of transparent materials. In solar thermal systems, AR coatings have been used in parabolic trough collectors, either in selective absorbers or glass envelopes that are positioned around the receiver tube.

The refractive index mismatch between a material and the air produces reflectance losses that can be reduced by applying an antireflection coating with an intermediate refractive index between air and the substrate material. An ideal homogeneous single-layer AR coating having an optical thickness of one-quarter of a wavelength will have its refractive index $n_c=(n_a n_s)^{1/2}$ (where n_a and n_s are the refractive indices of the air and the substrate, respectively) [6,7]. The refractive index of soda lime glass is roughly 1.54, which implies an ideal refractive index of 1.24 for an AR coating. The antireflection coatings can either be materials with an intermediate refractive index between the substrate and air [8,9] or porous coatings [10] or refractive index-graded materials [6,11,12]. Most widely used materials for AR coatings are dielectric materials such as SiO_2, alumina and titania with refractive indices of 1.45, 1.65 and 2.30 [1,8]. SiO_2 is the most ideal material due to its low refractive index, good durability and environmental resistance. Also, since glass is used for most of the applications, SiO_2 based AR coatings have strong adherence and hence is an added advantage.

AR coatings are developed by various approaches such as multilayers, porous nanostructures, coatings with nanoparticles, etc. The preparation of AR coatings can be achieved either by dry deposition techniques (CVD, PVD, etc.) or by wet techniques (spray coating, brush casting, spin coating, dip coating, etc.) and through sol–gel process. Wet deposition techniques are advantageous for the preparation of multi-phase materials since any non-volatile compound that is dispersed or dissolved into the solution will be homogeneously distributed in the coatings deposited. Dip coating is an ideal method to prepare thin layers from chemical solutions since it offers a good control on the coating thickness. The most important advantage of sol–gel process is the ability to tailor the microstructure of the deposited film, thereby inducing porosity

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into it. This is achieved generally by using pore-inducing polymers like polysiloxanes [2,4], nano-particles [7], functional block co-polymers [13], etc. Multilayer coatings have also been explored to achieve graded refractive index in the coating and to broaden the maximum transmittance for wide range of wavelengths in the visible region [6,14]. However, multilayer coatings by the sol–gel process are difficult to fabricate as it requires the optimization of optical properties and thicknesses of more than two layers. Hence, it is always preferred to have a deposition process which is simple, yet cost effective.

In this work, we have prepared nano-porous single layer AR coatings on glass substrates using tetraethoxysilane (TEOS). Polyethyleneglycol (PEG) and Triton X-100 were used as the polymeric porogens. The sol composition was optimized and the effect of thickness and heat treatment on the optical property of the AR coating was studied. The optimized AR coating showed a maximum transmittance of ~97.5% in the visible region. Sol stability over a long period is a very important aspect for the industrial scale production of the AR coating. The sol stability in turn affects the composition and thickness of the AR coating, which is essential for the reproducibility. This can be controlled mainly by withdrawal rate of the substrate and the viscosity of the sol. The former can be monitored, but the viscosity of sol changes with time on aging, depending on the degree of the hydrolysis of the alkoxide and subsequent polycondensation. Therefore, it is important that the viscosity remains constant in order to produce films with optimized thickness. We have hence studied the stability of the prepared sol. In addition, we have also studied the stability of the AR coating over a period of time (1 year). Conventionally, low boiling point (B.P.) solvents such as ethanol (B.P.=78.3 °C) are extensively used for the synthesis of the sol. These solvents, because of their higher evaporation rates, lead to non-uniform coatings with low transmittance even under normal industrial environment [15]. Hence, it is important to use high boiling point solvents for industrial scale production of AR coatings. 1-Methoxy-2-propanol (B.P.=120 °C) has been used in the present work for achieving reproducible and uniform AR coatings. Several advanced analytical characterization techniques were used to study the optical, mechanical and structural characteristics of the single layer SiO_2 coatings.

2. Experimental details

2.1. Materials

Tetraethoxysilane (TEOS) was used as the precursor for the SiO_2 coatings and was procured from Sigma Aldrich. 1-Methoxy-2-propanol was used as the solvent and was also obtained from Sigma Aldrich. PEG 200 and Triton X-100 were procured from Fluka. Nitric acid (HNO_3) was used for the acid catalysis of the sol and was from Merck Chemicals. Milli-Q water (H_2O) was used for all the aqueous solutions prepared. Microscopic glass slides (soda lime) were used for the coating deposition.

2.2. Synthesis of hybrid sol and coating deposition

A hybrid silica sol was prepared by mixing the precursor materials in the molar ratio: H_2O:1-methoxy-2-propanol:TEOS: HNO_3=1.0:2.0:0.35:0.005. Initially, 1-methoxy-2-propanol was mixed with the silica precursor (TEOS) in a sealed glass container by magnetic stirring for 5 min. Then an optimized amount of PEG (0.012 M) was added and stirred for 2 h, which resulted in a homogeneous transparent solution. To carry out hydrolysis of TEOS molecules, a mixture of nitric acid and Milli-Q water was added drop wise to the solution and stirred for 5 h followed by Triton X-100 (0.003 M). The molar ratio of TEOS:PEG:Triton X-100 was 20:4:1. The final mixture was magnetically stirred for 24 h. The transparent silica sol obtained was kept at room temperature for 3 days for aging and finally used for deposition.

Soda lime glass slides were cleaned by soaking them in soap solution for 15 min followed by thorough water cleaning. They were then rinsed with acetone and dried at room temperature. Coatings were deposited on the cleaned glass slides using a single dip coater (model SDC-2007C, Apex Instruments Co. Pvt. Ltd.). After the deposition of SiO_2 coatings, the glass substrates were immediately heat treated at 100 °C for 1 h and then at 400 °C for 2 h in the furnace. The thickness of the coating was optimized by varying the lifting speed of the substrate.

2.3. Characterization of the coating

UV–vis spectrophotometer (PerkinElmer, Lambda 750) was used to measure the transmittance of the coatings. The ellipsometric data for the films were measured in a spectroscopic phase modulated ellipsometer (Model UVISELTM 460, ISA JOBIN-YVON SPEX) in the wavelength range of 300–900 nm. The microstructural details of the coatings were investigated using atomic force microscopy (AFM-Bruker) and high-resolution field emission scanning electron microscopy (FESEM, model Supra 40 VP, Carl Zeiss). The 3D morphology of the coatings was also studied using NanoMap 500 LS 3D surface profilometer. Fourier transform infrared (FTIR) spectroscopic studies of the coatings were carried out using Bruker Vector 22 FTIR spectrometer to study the chemical structure of the coatings. The bonding structure of the coatings was characterized by X-ray photoelectron spectroscopy (XPS-SPECS) using non-monochromatic Al K_α radiation (1486.8 eV). X-Ray diffractometer (Bruker, D8 Advance) was used to study the structural characteristics of the antireflection coatings. Thermo-gravimetric analysis (TGA) of the coatings was carried out in the temperature range from room temperature to 1000 °C in an air atmosphere using PerkinElmer Instrument (Pyris 1, HT-TGA).

The hardness or scratch resistance of the prepared AR coatings was evaluated using Pencil Hardness Tester (model Elcometer 501) according to ISO 15184 standards. This test is a constant-load scratch test, which uses pencil leads of different hardness grades (9B–9H) as the scratch stylus. A fixed constant load with indenters (pencil leads) of different hardness values is applied on the samples. The hardest pencil grade that does not cause damage to the coating is taken as its pencil hardness. Adhesion of the coating with the substrate was assessed using the Cross Hatch Cut method (model Elcometer 107), according to the ASTM D3359 standards. A pressure-sensitive adhesive tape was applied over the cuts made at right angles to each other and pulled in a single smooth action. Adhesion was then assessed by comparing the fraction of coating removed from grid of squares against the ASTM standards.

3. Results and discussion

3.1. Optimization of single layer silica based antireflection coatings

Silica hybrid sol was prepared using PEG and Triton X-100 as discussed in Section 2.2. Optimization of PEG and Triton X-100 was very important to get a smooth coating. PEG and Triton X-100 are known to induce porosity in the coatings on thermal treatment. This lowers the refractive index of the coating and thus the AR properties are better. However, the homogeneity of the coatings was adversely affected by this factor and decreased with the PEG content. Therefore, the concentration of PEG was optimized at 0.012 M and Triton X-100 at 0.003 M. Concentrations lower than this resulted in poor transmittance of the coatings, whereas, higher

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