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Accelerated ageing tests of carbon nanotube spectrally selective solar absorbers

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ABSTRACT

A novel tandem type of spectrally selective solar absorber using a homogeneous multi-walled carbon nanotube (MWCNT) coating as absorbing layer has been fabricated. The MWCNT absorber was prepared by facile and efficient electrophoretic deposition and exhibited good spectral selectivity. To assess the durability of the MWCNT absorber, condensation and thermal stability accelerated ageing tests were performed according to the international standard ISO 22975-3: absorber surface durability. The primary results revealed that the MWCNT absorber had a great thermal stability but was not resistant to condensation since the porous MWCNT coating permits water migration through the pores down to the aluminum substrate which as a result oxidizes, confirmed by the analysis of Energy Dispersive Spectroscopy. Therefore, different types of thin films such as dense silica, silica-titania were deposited on top of MWCNT absorbers as protective layer to prevent the penetration of condensed water. Although all MWCNT absorbers coated with protective layer had little or no gain in spectral selectivity compared to those without protective layer, accelerated ageing tests indicated that the long-term durability was significantly improved. In thermal stability test, all protective layer coated MWCNT absorbers showed similar performance to the uncoated samples and had a negligible or even negative performance criterion (PC) value after 600 h testing. In condensation test, the obtained PC values were 0.002, 0.013 and 0.014 for silica, 70/30 and 50/50 silica-titania film coated MWCNT absorbers respectively. All the PC values were less than 0.015 after 600 h of accelerated ageing tests, which confirmed that the absorbers were qualified according to ISO 22975-3.

1. Introduction

This study is a continuation of earlier work where spectrally selective solar absorbers were fabricated using carbon nanotube (MWCNT) coating deposited by electrophoresis as absorbing layer on aluminum substrate [1]. Electrophoretic deposition (EPD) has been proven to be a facile and efficient method for preparing MWCNT coating on metal substrate. Compared to the traditional commercial vacuum deposition fabrication of spectrally selective absorbers, EPD is cheaper and more environmentally-friendly owing to the use of aqueous suspensions and low chemical consumption. In addition, there is no need for an inert atmosphere or vacuum environment during the final drying and heat treatment of the MWCNT coated absorbers. MWCNTs have a diameter of tens of nanometers which results in plasmon excitations [2] and improved absorption of solar radiation [3]. The thickness of the deposited MWCNT coatings is at a scale of hundreds of nanometers. Combined with the underlying highly infrared reflective aluminum substrate, the fabricated MWCNT absorbers exhibit good spectral selectivity with a solar absorptance of $>0.90\%$ and a thermal emittance of $<0.15\%$. While the method seems promising, the ability of the MWCNT absorbers to withstand deterioration caused by external environmental effects during normal operation has to be evaluated before further development or commercialization. For instance, increased temperature combined with condensation during the operation could accelerate many different kinds of chemical reactions, which leads to a higher rate of degradation of materials and consequently their performance. At high temperatures, the oxidization of the thin film absorber and/or the substrate surface is promoted, possibly resulting in a loss of spectral selectivity. Therefore, it is critical to assess the durability of solar absorbers in terms of the service life time. Although several articles have reported to use CNTs or CNT composite for spectral selective absorbers [4–6], no detailed results on

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accelerated ageing tests have been found. In this report, the results from thermal stability test and condensation test are presented. The tests followed the recommendations of the international standard ISO 22975 solar energy – collector components and materials – Part 3: Absorber surface durability [7].

To improve the durability of solar absorbers, a protective and anti-reflective layer can be added on top of the absorbing layer. This layer has to be dense enough to prevent penetration of condensed water so that the underlying surface is protected from oxidation or corrosion. Since the added layer could have an impact on the optical properties, it should be thin enough to avoid any increase in thermal emittance. Silica and silica-titania coatings have been employed as anti-reflection or self-cleaning layer in many different applications [8–12]. In this study, different silica and silica-titania formulations were tested as protective film against moisture and condensation.

This paper focuses on the investigation of long-term durability of MWCNT absorbers and protective film coated MWCNT absorbers, evaluated through accelerated ageing tests.

2. Sample preparation

The procedure of preparing MWCNT absorber samples was described in the author's previous paper [1]. Briefly, MWCNTs dispersed and stabilized in an aqueous solution containing a surfactant were deposited on aluminum substrates by electrophoresis. Thereafter the MWCNT coated samples were heat treated for solidification. These samples were then subjected to accelerated ageing tests or further processing of protective film.

Three types of protective films were investigated: silica film and silica-titania films with 70/30 and 50/50 of Si/Ti molar ratios. The sol-gel process was used to prepare these solutions which originated from the two references [13,14]. For silica film, Tetraethoxysilane (TEOS, $\geq 99\%$) as silica precursor was firstly mixed with ethanol ($\geq 99.5\%$) before DI-water containing HCl (37%) was added. HCl was used as catalyst for the hydrolysis of TEOS. The molar ratio of TEOS: EtOH: H_2O: HCl was equal to 1: 35: 5: 0.04. In order to hydrolyze TEOS, the resulting mixture was stirred for 2 h at room temperature before coating. For silica-titania sols, TEOS, ethanol, DI-water and HCl were mixed and stirred for 30 min. Then the mixture was diluted with ethanol before acetylacetone ($\geq 99\%$) and Tetrabutyl orthotitanate (TBOT, $\geq 98\%$) were sequentially added. The final molar ratios of TEOS: EtOH: H2O: HCl: TBOT: acetylacetone were 1: 66: 4: 0.08: 1: 1 for 50/50 silica-titania sol and 0.7: 30: 2: 0.04: 0.3: 0.3 for 70/30 silica-titania sol. The resulting sols were stirred for 6 h before coating process. All the chemicals used in these experiments were from Merck and had no pre-treatment.

A spin coater, Specialty Coating Systems SCS 6800, was employed to obtain silica / silica-titania coatings on top of MWCNT absorber with a size of 30 mm $\times$ 32 mm. A syringe containing 0.3 ml of precursor solution was ejected onto the center of sample surface. The solution spread and covered the surface in a fraction of a second. A subsequent 30 s spinning process allowed further evaporation of solvents and formation of a homogeneous silica or silica-titania coating. To control the thickness, spin speed was varied between 1500 – 3000 rpm and 3000–6000 rpm for silica and silica-titania coatings respectively. After the coating process, the protective film coated MWCNT absorbers were first dried for a few minutes at room conditions and then heat treated in a tube furnace under atmospheric environment. The rate of temperature increase was fixed to 50 °C per minute. The peak/final temperature T_p in the heat treatment was 400 °C and 500 °C for silica and silica-titania coatings respectively. The heating was turned off when T_p had been reached and the samples were left in the tube furnace until the temperature decreased to 300 °C before they were removed to room conditions for faster cooling.

3. Accelerated ageing tests

ISO 22975-3 accelerated ageing test procedure includes condensation, thermal stability and high humidity air containing sulfur dioxide tests. Only the first two tests are critical for the type of solar absorbers studied in this work.

A climate chamber VCL 4010 from Vötsch Industrietechnik was used to run condensation test. For humidification running, the temperature and the relative humidity in the inner chamber can be controlled in the ranges of from 10 to 95 °C and from 10% and 98%, respectively. The recommended sample temperature for condensation test is 40 °C, following the procedure presented in the standard [7]. This temperature was maintained by a circulating water pipe, which connected to a thermoregulator Techne TE-10D Tempette, running through the sample holder. In order to ensure condensation of water on the surface of the tested samples, the temperature and humidity in the inner chamber during the condensation test were set to 45 °C and 95% respectively for all samples. During the entire test, condensation droplets of water could be observed on the sample surface at all times. After specified test intervals, the samples were taken out of the climatic chamber and dried at room conditions before reflectance measurement.

Based on optical properties i.e. solar absorptance and thermal emittance and the corresponding maximum operation temperature T_{max}, ISO 22975-3 has suggested test temperatures for thermal stability test. For the MWCNT absorbers investigated in this work, T_{max} is between 186 and 190 °C, and the corresponding recommended test temperature is 259 °C. A slightly higher temperature of 265 °C was used for all the samples in this work. The thermal stability test was performed using a tube oven Entech, ESTF 40-120/11. Samples were placed in the tube before the oven temperature was ramped up at a rate of 50 °C per minute. The temperature was then kept constant at 265 °C until the end of each test period. After each test period, heating was turned off and the tested samples were removed from the oven at 100 °C for faster cooling to room temperature. Reflectance measurements were then performed for the assessment of durability.

Both condensation test and thermal stability test were carried out in specified test intervals of 150, 300 and 600 h. For each ageing test and test duration, two samples with similar solar absorptance and thermal emittance were used.

4. Characterization

Surface morphology and atomic composition were investigated using a ZEISS Merlin VP Scanning Electron Microscope equipped with an Energy Dispersive Spectroscopy (EDS) system from Oxford Instruments for element analysis. An automated angle M-2000FI spectroscopic ellipsometer system from J. A. Woollam Co. was employed to measure the optical constants of the MWCNT coating.

The spectral reflectance of all the samples was measured in the wavelength range of 0.3 – 20 μm. A PerkinElmer Lambda 950 UV/vis spectrometer equipped with an integrating sphere of diameter 150 mm was used for reflectance measurement from 0.3 to 2.5 μm. For the infrared wavelength range 2.0–20 μm, the samples were measured using a Bruker Tensor II FT-IR spectrophotometer. The measurement results were used to calculate normal solar absorptance, α, theoretically defined as a weighted fraction of the absorbed radiation to the incoming solar irradiation on a surface,