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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 × 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,

$$\alpha = \frac{\int_{0.3}^{2.5} I_{sol}(\lambda)(1 - R(\lambda))d\lambda}{\int_{0.3}^{2.5} I_{sol}(\lambda)d\lambda} \tag{1}$$

and normal thermal emittance, ε, i.e., the ratio of emitted radiation of a surface to that of Plank's blackbody, I_p, at 100 °C,

$$\varepsilon = \frac{\int_{2.0}^{50} I_p(\lambda)(1 - R(\lambda))d\lambda}{\int_{2.0}^{50} I_p(\lambda)d\lambda} \tag{2}$$

where I_{sol} is the spectral solar irradiance defined according to ISO standard 9845-1 (1992) for air mass of 1.5. λ is the wavelength of incident radiation in the unit of μm and $R(\lambda)$ is the reflectance at a certain wavelength. An extrapolation method was introduced to estimate the reflectance from 20 to 50 μm so that the calculation by Eq. (2) can be completed. The standard deviation of α and ε, which has been calculated from repeated reflectance measurements of the same sample over an extended time period, is respectively 0.002 and 0.02 for the solar absorber samples in this study.

The durability of absorber surface was assessed by the following performance criterion (PC) [7]:

$$PC = -\Delta\alpha + 0.5\Delta\varepsilon$$

where Δα is the change in solar absorptance and Δε is the change in thermal emittance after testing. The 0.5 factor reflects the lesser importance of a change in thermal emittance compared to a change in solar absorptance. In this study, the qualification procedure, which is little different from the description in the standard, is: if PC $>$ 0.05 after 150 h of testing, the absorber surface is disqualified; If PC $\leq$ 0.015 after 600 h of testing, then it is qualified. The threshold value of 0.05 means that the optical performance of absorber surface during the service life time is expected to reduce to no more than 95% of its original value. It is based on a design service life time of 25 years. For intermediate PC values, additional testing with at different test temperatures and durations is needed to determine whether or not the absorber surface is qualified. It is worth noting that a negative PC value would indicate a factual improvement of the spectral selectivity over service life time.

5. Results and discussion

5.1. Accelerated ageing tests without protective films

Fig. 1 presents reflectance spectra of MWCNT absorber samples before and after subjection to accelerated ageing tests. The test time was 150 h. After the thermal stability test, the reflectance was nearly unchanged, i.e. the solar absorptance and thermal emittance remained identical (Fig. 1a). This was expected since the MWCNT coatings were heat treated at 500 °C in air prior to the thermal stability test and consequently should be thermally stable at the much lower test temperature of 265 °C. However, condensed water on the sample surface had a serious impact on the spectral reflectance. After 150 h of testing, solar absorptance did not change and remained at 0.91 while the peak reflectance occurring in the high intensity region of the solar spectrum was reduced and the reflectance in the near infrared range increased. However, the reflectance in the far infrared range decreased significantly (Fig. 1b). The resulting thermal emittance increased from 0.16 to 0.86 after the condensation test. The spectral selectivity was worsened drastically and the resulting PC value of 0.35 disqualified the absorber. The increased absorption in the infrared range is in all probability caused by the oxidation of the underlying aluminum substrate [15]. This is confirmed by the EDS analysis, presented in Figs. 2 and 3. The oxygen content of the MWCNT absorber after condensation test (Fig. 2) was much higher than that of the MWCNT absorber before condensation test (Fig. 3). Due to the porosity of MWCNT coating, the condensed water was able to pass through the coating to form an aluminum hydroxide or oxo-hydroxide through oxidation. The rate of oxidation was accelerated by the elevated test temperature of 40 °C. It is very unlikely that the condensation test oxidizes the MWCNTs themselves. Usually strong acidic conditions need to be present in order for that to take place [16]. The surface of the samples appeared to be slightly rougher after the condensation test. This resulted in more light scattering which could explain the increase in absorption in the visible wavelength range, as shown in Fig. 1b. In order to improve the long-term durability of the MWCNT absorber, the oxidation of aluminum substrate has to be minimized. One of the simplest ways would be to add a dense coating on top of the MWCNT layer.

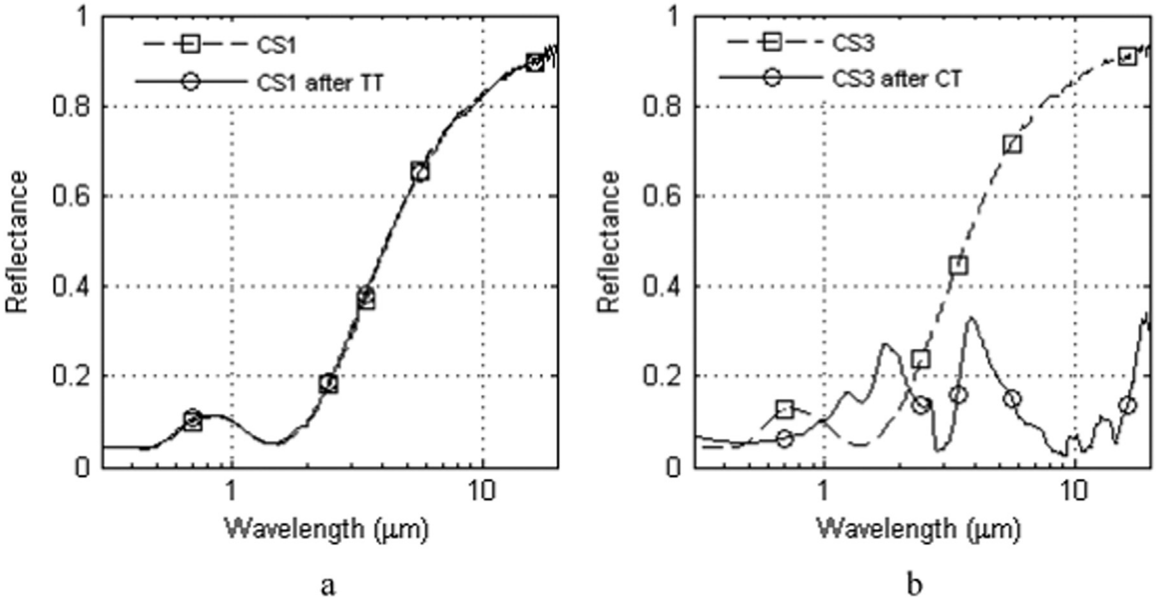


Fig. 1. Reflectance spectra of MWCNT absorbers before (dashed curves) and after (solid curves) 150 h accelerated ageing test: a) Thermal stability test; b) Condensation test.

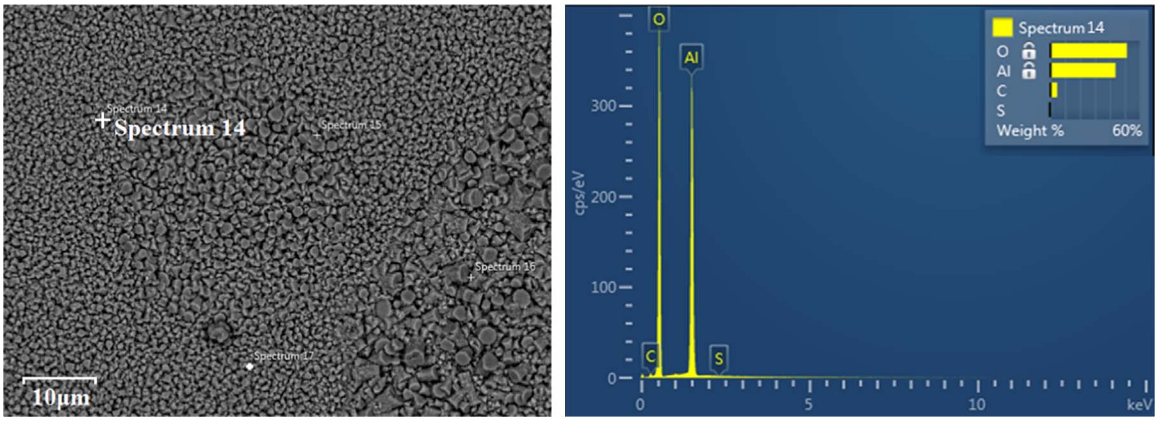


Fig. 2. SEM image (Right) and EDS result (Left) of MWCNT absorber surface without protective film after condensation test.

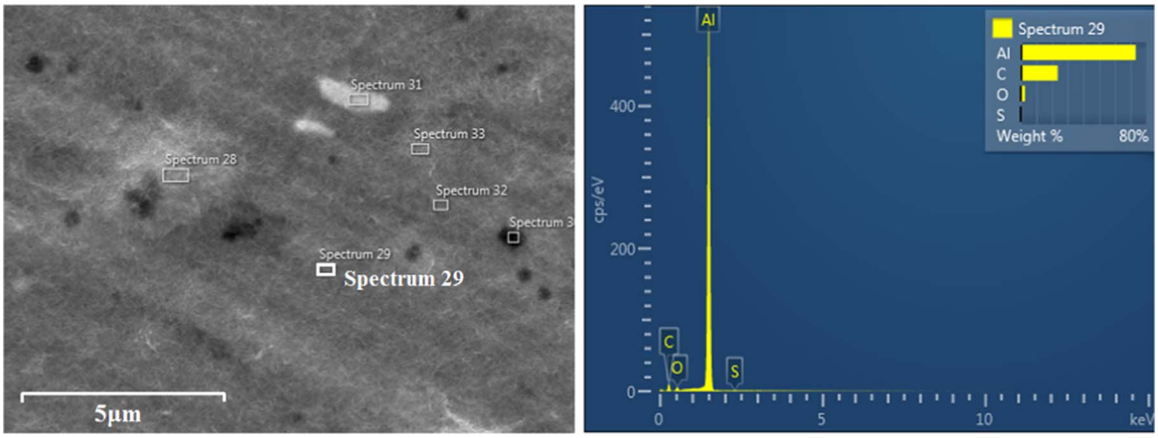
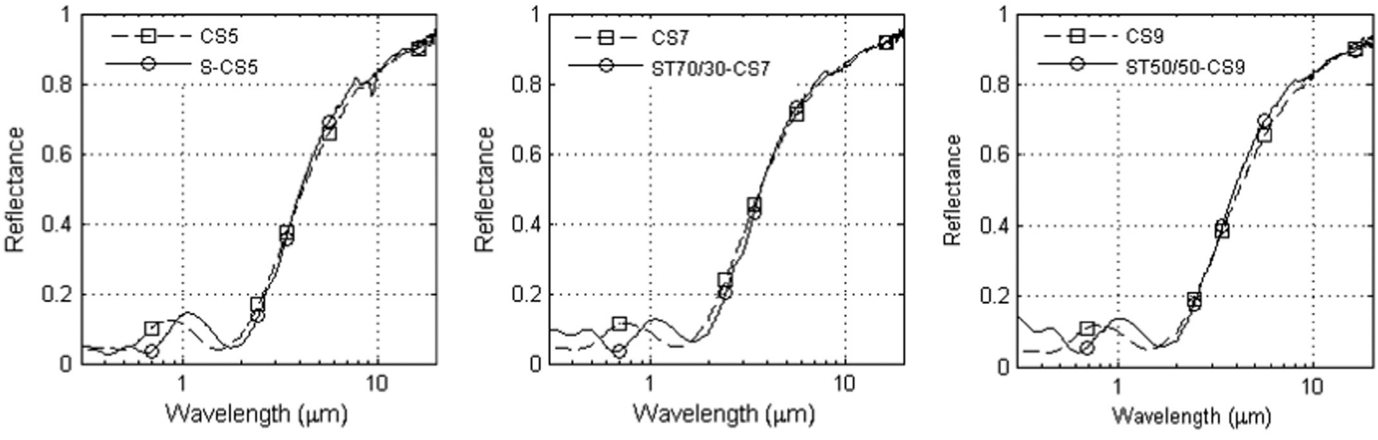


Fig. 3. SEM image (Right) and EDS result (Left) of MWCNT absorber surface without protective film before condensation test.

5.2. *Protective film influence on reflectance*

While a protective film shields the MWCNT coating from the penetration of condensed water, the protective film also affects the optical properties of the MWCNT absorber. The three tested protective films of silica, 70/30 silica-titania and 50/50 silica-titania have different refractive indexes [17] and thus the effect on spectral reflectance of the absorber samples differs from each other. The thickness of the protective films is the other decisive factor affecting the spectral reflectance. An optimized film thickness would result in a destructive interference trough at high intensity solar irradiation wavelengths, reducing the reflectance and hence increasing the solar absorptance. During the experiments, each type of film was coated with various thicknesses in order to examine the effect. The spectral reflectance of the samples before and after coating with an optimized thickness is shown in Fig. 4 for each type of protective film. With the optimized thickness of silica film, the minimum reflectance point moved from the wavelength of 0.9 µm to 0.7 where it falls into the region of significant solar irradiance. This change increased the overall



a b c

Fig. 4. Reflectance spectra of MWCNT absorbers before (dashed curves) and after protective coating (solid curves): a) Silica; b) 70/30 Silica-titania; c) 50/50 Silica-titania.

Table 1
Solar absorptance and thermal emittance without and with protective films.

Sample type	Silica film		70/30 Silica-titania film		50/50 Silica-titania film	
	α	ε	α	ε	α	ε
Without protective film	0.92	0.17	0.92	0.15	0.91	0.18
With protective film	0.93	0.17	0.92	0.15	0.91	0.17

absorption within the solar spectrum which resulted in a rise of solar absorptance from 0.92 to 0.93 (see Table 1). The thermal emittance maintained the same value of 0.17 after coating with a silica film, which indicates that the silica film is thin enough and at the order of 100 nm [18].

The two types of silica-titania films have similar effects. Although a destructive interference occurred at the wavelength close to the high intensity region of the solar spectrum, the overall absorption was not enhanced due to the increased reflectance in other regions of the solar spectrum. The resulting solar absorptance remained unchanged after the application of the protective films. The effect of coating on the spectral reflectance in the infrared region was nearly negligible.

The reason for the minor anti-reflective (AR) effect of the protective coatings is likely due to bad refractive index matching. The real part of the refractive index of the MWCNT coating was estimated using Elliopsometry to be 1.38 at a wavelength of 550 nm. However, this value is highly uncertain since the ellipsometer was struggling with getting accurate refractive index estimations because of the high surface roughness of the MWCNT absorber. A previous optical study of single-walled CNT (SWCNT) films reported a real part refractive index value at 550 nm of 1.01 and 1.58 for a respectively, low volume fraction and high volume fraction SWCNT coating [19].

An optimal AR coating should have a real part of the refractive index equal to the square root out of the underlying material, which in this case would be 1.17. It is very difficult to find dense protective materials that could work as an AR film with such low refractive index. The material with the lowest refractive index of the used protective coatings in this study is the pure silica, which has an index of 1.4 [17].

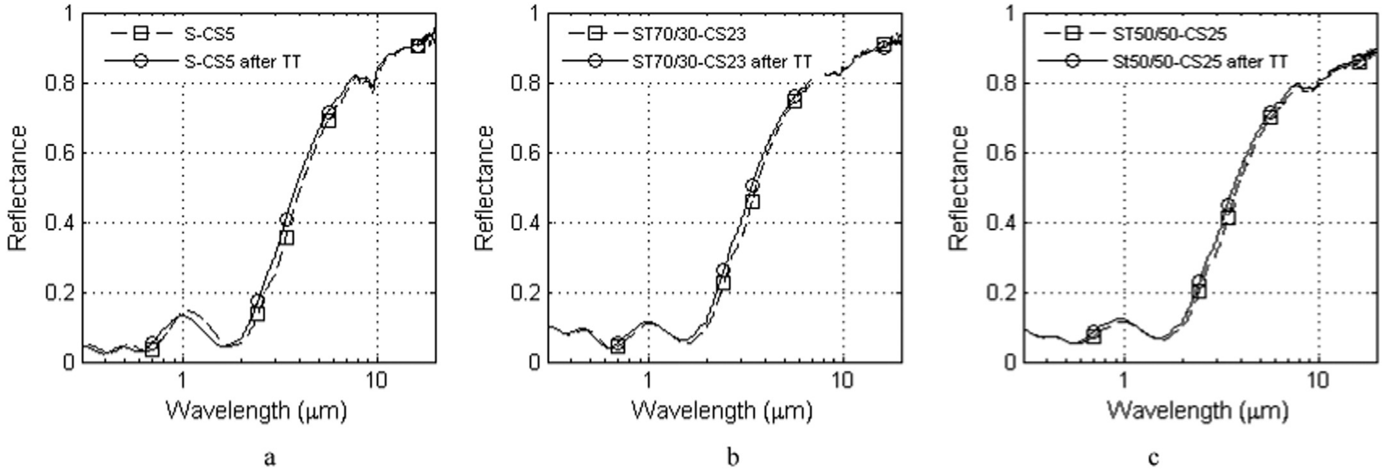
5.3. *Accelerated ageing tests with protective films*

MWCNT absorber samples coated with protective films were subjected to thermal stability test. The reflectance was measured after 150, 300 and 600 h of testing. Similar to the samples without protective films, the spectral reflectance remained almost unaffected in the UV-Vis-NIR spectrum for all the protective film coated samples after the test. There was a slightly reduced thermal emittance due to higher reflectance in the infrared range i.e. little improvement on spectral selectivity, shown Fig. 5. The PC values attained after 600 h were -0.002, -0.007 and 0.002 for MWCNT absorbers coated with silica, 70/30 silica-titania and 50/50 silica-titania respectively. The negative values indicate a small improvement of performance after 600 h of thermal stability testing.

Condensation test of the coated samples also gave favorable results. As Fig. 6 shows, the transition from low to high reflectance of the samples exhibited a shift to red after 600 h testing, more clearly for the silica-titania films coated samples than the silica coated samples. As a result, there was a little increase in the thermal emittance. For silica coated MWCNT absorbers, the computed PC value was 0.002, while it was 0.013 and 0.014 for 70/30 and 50/50 silica-titania films respectively after 600 h testing (Table 2). Although the positive PC values indicate a minor degradation over a long period due to the increased thermal emittance, all the values are lower than 0.015 and therefore no further testing is needed. Hence, the protective film coated MWCNT absorbers are considered as qualified if the adhesion requirements are met which is outside the scope of the current work.

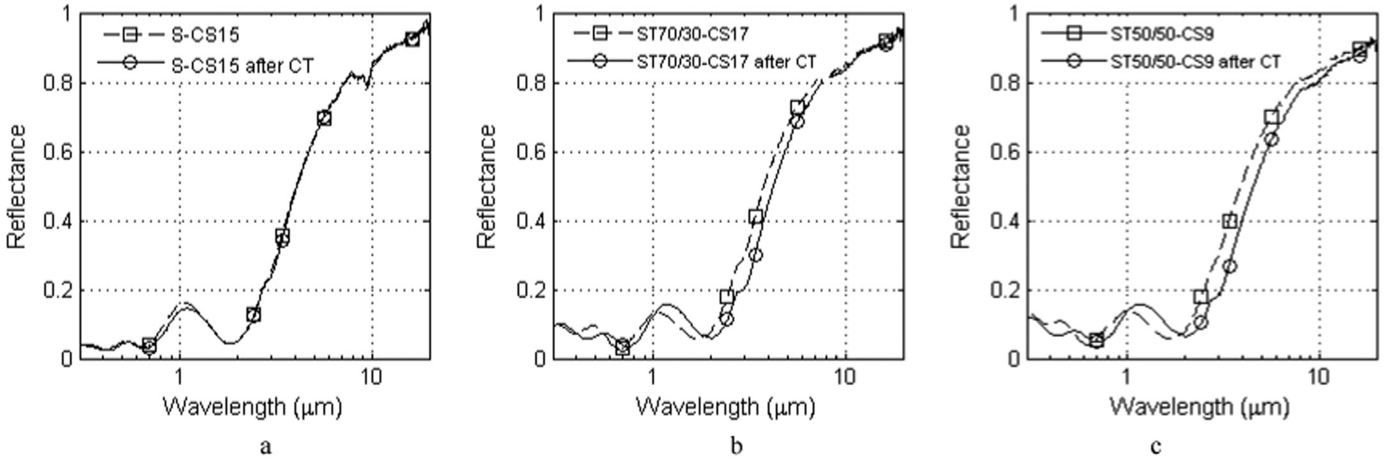
6. Conclusions

In this study, accelerated ageing tests were performed on novel carbon nanotube (MWCNT) spectrally selective solar absorbers. Although they exhibited excellent thermal stability, the thermal emittance of the MWCNT absorbers increased significantly already after 150 h of condensation testing and were therefore disqualified to meet the performance requirements for long-term durability. The cause is believed to be condensed water penetrating the porous MWCNT coating and reacting with the underlying aluminum substrates, causing aluminum oxidation products that absorb in the infrared. In order to improve the long-term durability of the MWCNT absorbers, three different protective films: silica, 70/30 silica-titania and 50/50 silica-titania were coated on top of MWCNT coating to protect it from direct contacting with condensation. The experiments have shown very promising results. With these protective films, the solar absorptance and the thermal emittance of the MWCNT absorbers were almost not affected at all.



a b c

Fig. 5. Reflectance spectra of MWCNT absorbers coated with protective films before (dashed curves) and after (solid curves) thermal stability test: a) silica; b) 70/30 silica-titania; c) 50/50 silica-titania.



a b c

Fig. 6. Reflectance spectra of MWCNT absorbers coated with protective films before (dashed curves) and after (solid curves) condensation test: a) silica; b) 70/30 silica-titania; c) 50/50 silica-titania.

Table 2
Solar absorptance, thermal emittance and PC values of condensation tested samples.

Sample type	Silica film			70/30 Silica-titania film			50/50 Silica-titania film		
	α	ε	PC	α	ε	PC	α	ε	PC
Before condensation test	0.929	0.167		0.919	0.150		0.910	0.172	
Tested for 150 h	0.932	0.172	−0.001	0.920	0.165	0.007	0.912	0.195	0.010
Tested for 300 h	0.932	0.172	−0.001	0.920	0.176	0.012	0.912	0.198	0.011
Tested for 600 h	0.932	0.177	0.002	0.920	0.178	0.013	0.912	0.204	0.014

In the thermal stability tests, all the protective film coated samples performed as well as the uncoated samples. After 600 h of condensation testing, the PC values, which were 0.002, 0.013 and 0.014 for silica, 70/30 silica-titania and 50/50 silica-titania respectively, were all lower than the limit of 0.015. This means that no further testing was needed and that the protective film coated MWCNT absorbers are all qualified in accelerated ageing tests. However, the purely silica coated samples performed the best and this material is consequently recommended as protective coating for the MWCNT solar absorbers.

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