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Development of High Absorption, High Durability Coatings for Solar Receivers in CSP Plants

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Abstract. In concentrated solar power systems (CSP), solar receivers need to have high light absorption and high durability at elevated temperatures. In this study, we preset high absorption, high durability coatings for solar receivers in CSP plants. Black pigments and TiO₂ porous structure were created to enhance optical properties, while the coating film was formed with 3 layers (base layer, absorption layer and top coating) to enhance durability. It is shown that this coating is stable at 850°C after 100 hours of aging, while keeping a solar-weighted absorptance value of 96-97%. This is higher absorptance and higher durability than conventional coatings used for CSP.

INTRODUCTION

In concentrated solar power systems (CSP), solar receivers need to have high light absorption and high durability at elevated temperatures. Nano Frontier Technology (NFT) has previously developed coatings composed of carbon nanotubes (CNTs) and TiO₂ with high absorption (99%); however, CNTs oxidise and burn above 500°C. In this study, CNTs were replaced with black pigments to enable working temperatures above 500°C which are suitable for solar receivers. Furthermore, a porous structure was created to enhance optical properties, while the coating film was structured with 3 layers (base layer, absorption layer and top coating) to enhance durability. It is shown that this coating is stable at 850°C after 100 hrs of aging, while keeping a solar-weighted absorptance value of 96-97%.

EXPERIMENTAL DETAILS

Sample Preparation

Organic titanium polymer, organic solvent and black pigments were mixed by ultra-sonication for 15 minutes to form a base layer that increases the adhesion between coatings and the substrate (stainless steel 253MA). The black pigments are complex metal oxides, and the diameter of each particle is approximately 700nm. To create an absorption layer ink, the titanium precursor is reacted with acetylacetone heated at 80°C for 6 hours and then diluted with 2-propanol. Liquid ink and black pigments were mixed by ultra-sonication for 30 minutes. An absorption layer is formed with pores that are expected to increase light absorbance. A tetraethyl orthosilicate mixture with organic solvent was used for the top layer to increase the durability of the coating.

Application of the Coating

To create a coating on a 3x3cm coupon, the substrate was first sprayed for 2 seconds with a base layer of an organic titanium polymer and black pigments at room temperature and then heated to 400°C for 30 minutes. The thickness of the base layer is approximately 8µm. To form the absorption layer, the liquid ink was sprayed on the base layer, which was held at 350°C. The titanium precursor solidifies the black pigments when it is crystallized by pyrolysis on the heater, creating pores by evaporation of the organic solvent. This process was repeated multiple times keeping a constant surface temperature. Controlling the temperature is key to creating the porous structure. Other crucial controls include the diameter of the spray nozzle, the gauge pressure of liquid ink, the gauge pressure of air, the distance to the substrate and the spraying time which may depend on the spraying machine. The sample is sprayed multiple times over at least a 2-minute period, but usually the process is completed within 5 minutes. The thickness of absorption layer is approximately 17µm. The coupon was then removed from the hotplate, and the top layer spray coated for 2 seconds at room temperature. Finally, curing was conducted for 30 minutes at 400°C. The thickness of the top layer is approximate 5-10nm. The thickness of three layers (base layer, absorption layer and top coating) is approximate 25µm. 8 samples were prepared for aging tests.

Aging Tests and Absorptance Measurements

Isothermal Heating

Aging tests and absorptance measurements were performed at the Australian National University (ANU). Three temperatures of 600°C, 750°C and 850°C and two aging times of 10hrs and 100hrs were chosen for the test in order to compare with measurements previously conducted at ANU for Pyromark [1]. The samples (coating + substrate) were aged for these durations in a programmable muffle furnace. Note that the heating and cooling rates were 3°C/min, hence the time to reach the target temperature and return to room temperature at the end of the process is additional to the aging time.

Spectral Absorptance Measurement

Before and after aging, the spectral reflectance of the sample was measured at room temperature using a spectrophotometer (Perkin Elmer UV/VIS/NIR Lambda 1050), at an incident angle of 8°. The spectrophotometer was equipped with an integrating sphere that measures the spectral hemispheric directional reflectance (HDR), or reflectance ρ , from the surface of the sample. As the samples are opaque, there is no transmittance and hence $\rho(\lambda) + \alpha(\lambda) = 1$, where α is the absorptance and λ is the wavelength. The spectral values were obtained with intervals of $\Delta\lambda = 10\text{nm}$. The standard G173-03 of the American Society for Testing and Materials (ASTM) for the spectral solar irradiance $G(\lambda)$ [2] was used to obtain the overall absorptance relative to solar irradiation, or solar-weighted absorptance α_s . In this study, α_s is defined as

$$\alpha_s = \frac{\int_{280\text{nm}}^{2500\text{nm}} \alpha(\lambda)G(\lambda)d\lambda}{\int_{280\text{nm}}^{2500\text{nm}} G(\lambda)d\lambda} \quad (1)$$

This integral was evaluated by a midpoint approximation of the rectangle method. The lower limit in Eq. (1) was set to 280nm to correspond to the lower bound of the G-173 data. The measurement of absorptance was done at room temperature, i.e. approximately 25°C. Based on studies done for other high-temperature coatings (e.g. Pyromark) we do not expect the absorptance to be significantly dependent on temperature [5].

RESULT AND DISCUSSION

Absorptance

Pristine State

The spectral absorptance of 8 coated samples in pristine state is shown in Figure 1. The difference in spectral absorptance is less than 0.8% in the wavelength range $400 \leq \lambda \leq 2500\text{nm}$, which is the region of interest since the spectral solar irradiance is higher at these wavelengths.

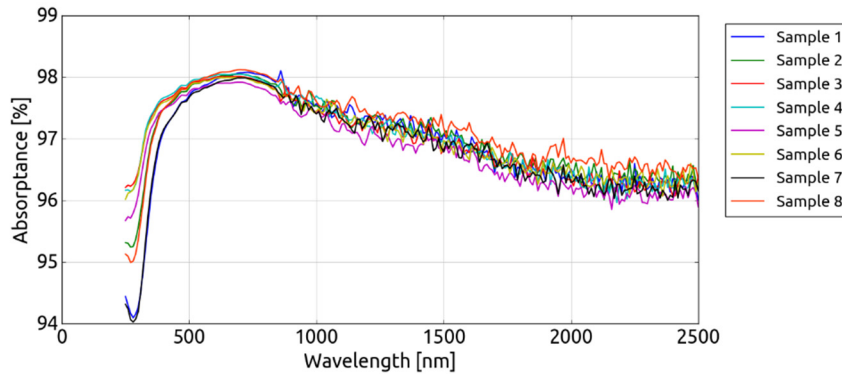


FIGURE 1. Spectral absorptance of samples in pristine state.

The spectral absorptance results for the coated Sample 8 in pristine state is shown in Figure 2 with colored triangles. The results are compared with measurements of spectral absorptance of a Pyromark coated sample from earlier testing [1], with the same SS253MA substrate. As shown in this figure, the spectral absorptance of the NFT coating is significantly higher than that of the Pyromark. Here, we report a solar-weighted absorptance for pristine samples of $96.93\% \pm 0.07\%$. We attribute the difference (or uncertainty) to variations in the coating itself.

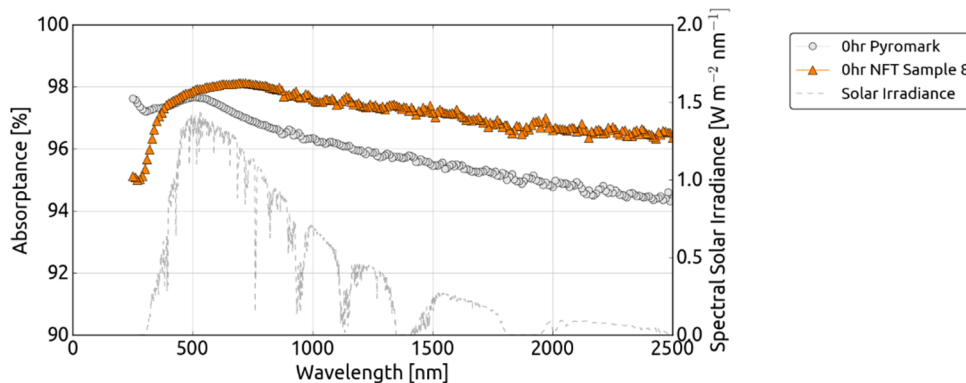


FIGURE 2. Spectral absorptance of coatings on SS253MA in pristine state and comparison with a Pyromark coated sample on the same substrate. The spectral solar irradiance is included for reference (right axis).

Absorptance for 600°C Aging

Samples 7 and 6 were aged at 600°C for a total time of 10 and 100 hours, respectively. Sample 6 was first aged for 10 hours, after which the absorptance was measured in room temperature, and then the same sample was aged for a further 90 hours, and absorptance re-measured. The absorptance as a function of wavelength is shown for both Sample 7 (Fig. 3a) and Sample 6 (Fig 3b). Here, and in all subsequent plots, the spectral solar irradiance is indicated

for reference (secondary axis). Both Sample 7 and Sample 6 exhibit a larger drop in optical performance than Pyromark at lower wavelength. However, for wavelengths larger than 700nm, the spectral absorptance did not exhibit noticeable changes while remaining significantly higher than Pyromark [4], whose value are included in these and subsequent plots as reference. The solar-weighted absorptance values are listed in Table 1. In all cases absorptance changes from the pristine state are small (within $\pm 0.1\%$), within the expected uncertainty bounds. Overall, the durability of Sample 7 and 6 was excellent for temperature of 600°C for the aging periods measured.

TABLE 1. Solar-weighted absorptance for the samples aged at 600°C

Time [hours]	0 (pristine)	10	100
Pyromark (9.25 μm), α [%] (change from 0)	96.55	96.54 (-0.01)	96.45 (-0.10)
Sample 7, α [%] (change from 0)	96.84	96.74 (-0.10)	-
Sample 6, α [%] (change from 0)	96.94	96.96 (+0.02)	96.89 (-0.05)

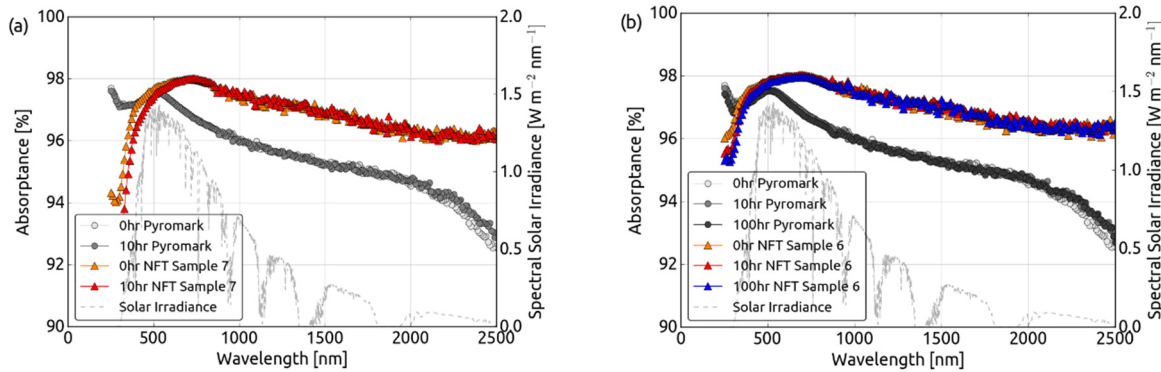


FIGURE 3. Spectral absorptance of coatings on SS253MA in pristine state, and after aging at 600°C with (a) Sample 7 aged for 10 hours and (b) Sample 6 aged for 10 hours and 100 hours (10 + 90 hours). Results for Pyromark (thickness 9.25 μm) are included as reference.

Absorptance for 750°C Aging

Following the same procedure as above, Sample 5 and 4 were aged at 750°C of isothermal aging for a total time of 10 and 100 hours, with Sample 4 first subject to 10 hours of isothermal heating and then an additional 90 hours to obtain absorptance measurements at 10 and 100 hours. The spectral absorptance results are shown in Figure 4, and the solar-weighted absorptance in Table 2. The results are similar to the 600°C case, although with perhaps a more noticeable drop in absorptance from pristine (-0.19%) after 100 hrs aging. The improvement compared to Pyromark is more pronounced after aging at 750°C compared to the 600°C case, as shown in Tables 1 and 2.

TABLE 2. Solar-weighted absorptance for the samples aged at 750°C

Time [hours]	0 (pristine)	10	100
Pyromark (9.5 μm), α [%] (change from 0)	96.66	96.47 (-0.19)	94.86 (-1.80)
Sample 5, α [%] (change from 0)	96.83	96.74 (-0.09)	-
Sample 4, α [%] (change from 0)	96.99	96.88 (-0.11)	96.80 (-0.19)

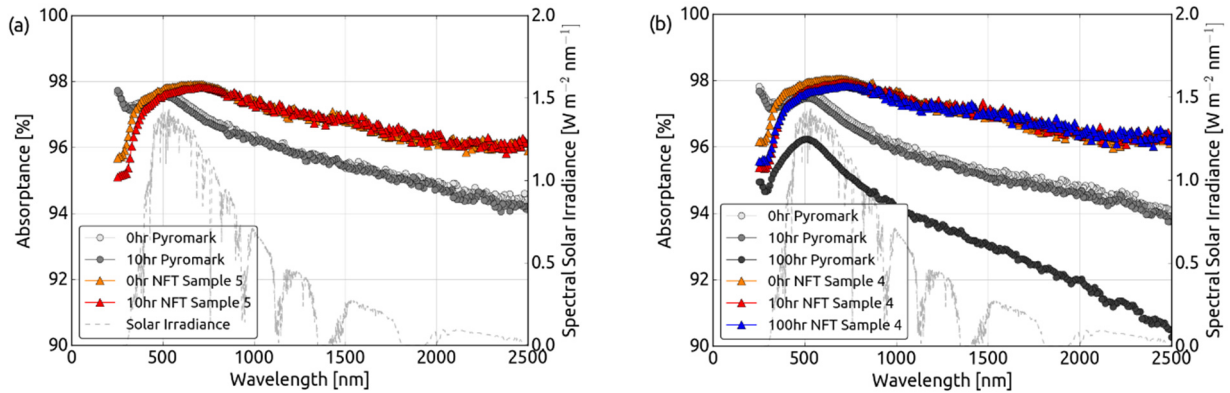


FIGURE 4. Spectral absorbance of coatings on SS253MA in pristine state, and after aging at 750°C with (a) Sample 5 aged for 10 hours and (b) Sample 4 aged for 10 hours and 100 hours (10 + 90 hours). Results for Pyromark (thickness 9.5µm) are included as reference.

Absorbance for 850°C Aging

In previous testing, high temperature aging at 850°C caused failure in Pyromark coatings on the SS253MA substrate [1]. Therefore, three NFT samples were prepared for the aging tests at 850°C. Samples 3, 1 and 2 were aged for a total time of 10, 20, and 100 hours respectively. Sample 1 was first subject to 10 hours of heating and then an additional 10 hours. Sample 2 was first subject to 10 hours of heating and then an additional 90 hours. The spectral absorbance measurements for Sample 3 and 1 are shown in Figure 5, Sample 2 in Figure 6, and the solar-weighted absorbance results in Table 3. These results clearly show a more pronounced drop of absorbance than for aging temperatures of 600°C and 750°C, particularly below around 1500 nm where the absorption is clearly lower. It is interesting to note there is little difference between the absorption drop after 20 hours (0.49%) and 100 hours (0.50%), indicating that the coating may have stabilized. However longer duration testing is necessary to confirm this. Once again, the drop in absorbance is significantly lower than for Pyromark, whose solar-weighted absorbance dropped by 2.75% from 96.77% in its pristine state to 94.02% after 10 hours, and finally failed after 100 hours. These results suggest that the NFT-developed coatings are not only better light absorbers but also more durable than Pyromark coatings.

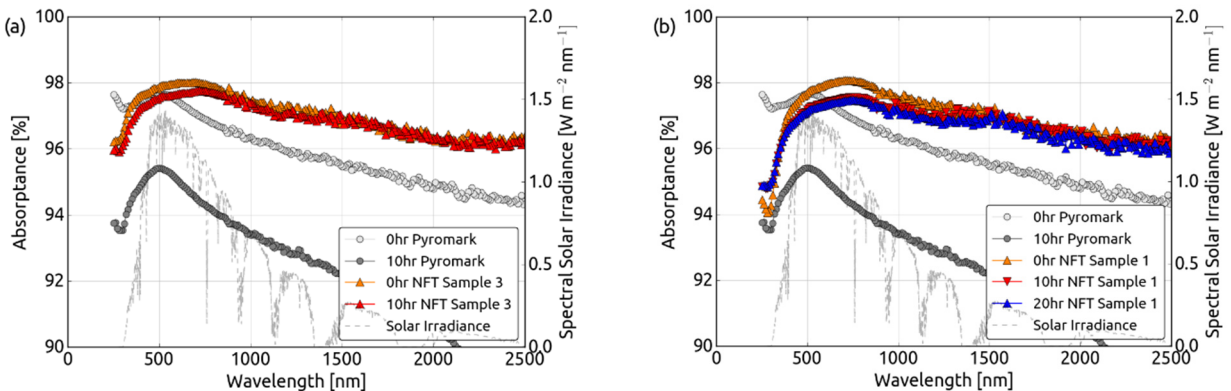


FIGURE 5. Spectral absorbance of coatings on SS253MA in pristine state, and after aging at 850°C with (a) Sample 3 aged for 10 hours and (b) Sample 1 aged for 10 hours and 20 hours (10 + 10 hours). Results for Pyromark (thickness 25µm) are included as reference.

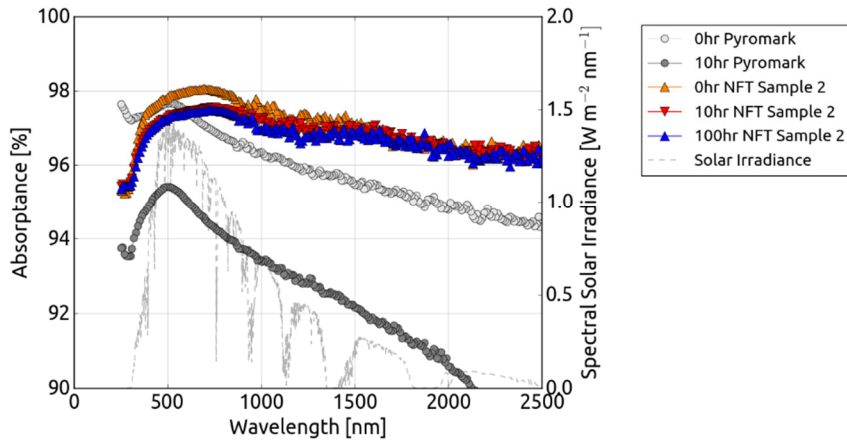


FIGURE 6. Spectral absorbance of coatings on SS253MA in pristine state, and after aging at 850°C with Sample 2 aged for 10 hours and 100 hours (10 + 90 hours). Results for Pyromark (thickness 25µm) are included as reference.

TABLE 3. Solar-weighted absorbance for the samples aged at 850°C

Time [hours]	0	10	20	100
Pyromark (25 µm), α [%] (change from 0)	96.77	94.02 (-2.75)	-	failure
Sample 3, α [%] (change from 0)	96.94	96.72 (-0.23)	-	-
Sample 1, α [%] (change from 0)	96.91	96.49 (-0.42)	96.42 (-0.49)	-
Sample 2, α [%] (change from 0)	96.96	96.54 (-0.42)	-	96.47 (-0.49)

Structure

This coating material was composed of black pigments and TiO₂. The black pigment coated with the titanium precursor bonds firmly when titanium crystallized by pyrolysis. Micro-structure pores, such as those shown in the scanning electron microscope (SEM) images in Figure 7, were created by spraying multiple times. These pores are thought to improve spectral absorbance, as suggested by our results.

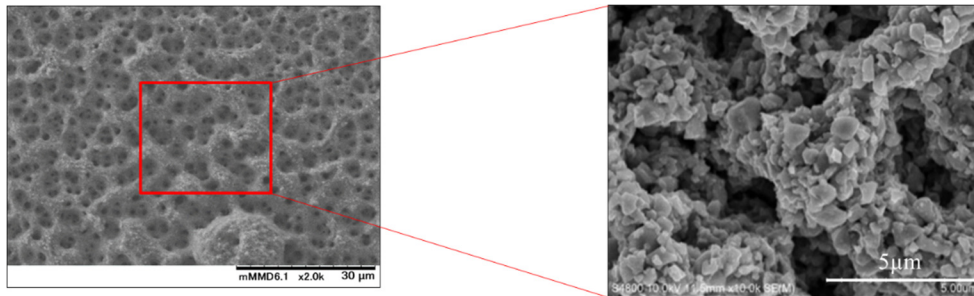


FIGURE 7. SEM images of the surface of coating. The black pigment is bonded by the TiO₂.

After 100 hours aging at 600°C, no considerable changes were observed in comparison to the pristine state. For the 750°C case, Figures 8 (a) and (b) show the SEM images of Sample 4 in its pristine state and after 100 hours

aging. No considerable changes were observed after aging. Some cracks were observed under 2000x magnification as shown in Figure 9(c). These cracks are thought to be a factor contributing to the drop in optical efficiency. The SEM analysis did not yield any evidence of crystallization.

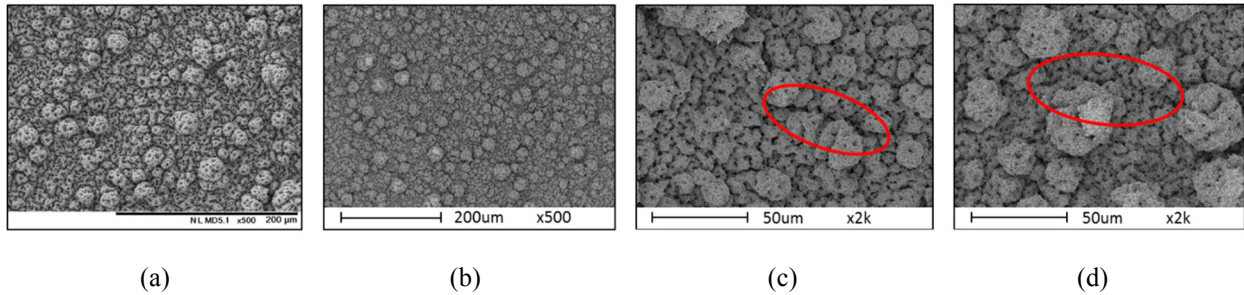


FIGURE 8. SEM images of Sample 4 in (a) pristine state (b-d) after 100 hours of aging at 750°C. Figures (c-d) show the same sample as (b) but at higher magnification. Some cracks observed are encircled by ellipses.

The morphology of the coatings is shown in Figure 9 and 10 for aging at 850°C. Cracking and crystallization were observed. There are multiple cracks in all samples aged at 850°C as shown in Figure 9(b-d) in comparison to the pristine images shown in Figure 9(a). We hypothesize that cracks are a contributing factor in the initial drop of optical efficiency, and that solar-weighted absorptance is lower for samples with larger cracks. These cracks are seemingly growing as the aging progresses, becoming a potential factor for failure in tests with a longer aging time. Regarding crystallization, no clear evidence of crystals was found after the first 10 hours of aging, whereas initial signs of crystallization were visible after 20 hours, as shown in Figure 10(a), and fully formed crystals were observed after 100 hours, as shown in Figure 10(b). Crystallization could become a potential factor for absorption drop and even failure in longer aging times.

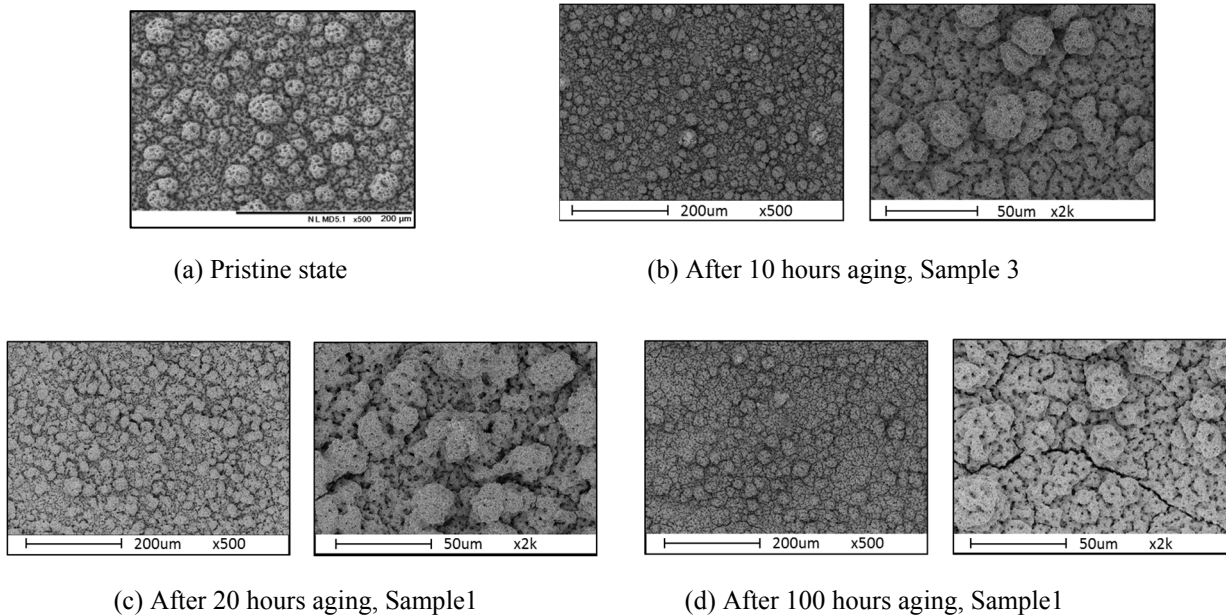


FIGURE 9. SEM images of Sample 3 in pristine state (a), after 10 hours of aging at 850°C (b), after 20 hours aging at 850°C (c) and after 100 hours of aging at 850°C (d). The cracks are clearly visible in the larger magnification images of 2,000x in (b), (c) and (d).

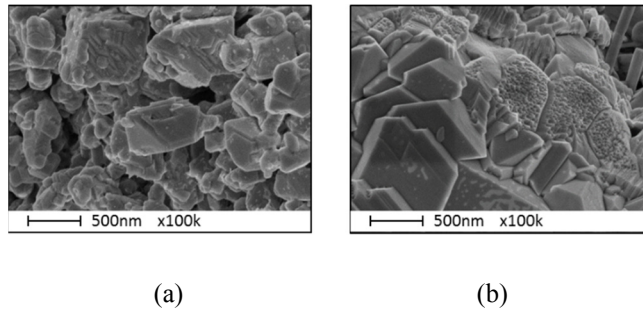


FIGURE 10. SEM images of (a) Sample 1 after 20 hours displays initial signs of crystallization, and in (b) Sample 2 after 100 hours crystals were clearly observed.

CONCLUSION

In this study, we develop a novel high-temperature absorber coating and investigated the effects of isothermal aging on optical performance and durability which is a basic requirement for CSP applications [3]. The proposed coating is composed of black pigments and TiO₂ has a porous structure that is thought to improve light trapping. After an isothermal aging for 100hrs at elevated temperatures, the coatings show a higher absorptance and a higher durability than the Pyromark coating conventionally used in many CSP applications. A good durability refers to consistency of appearance, lack of deterioration and/or delamination. However, aging at a temperature of 850°C results in cracking (in microscale) and crystallization (in nanoscale). These morphological changes could be a contributing factor the larger drop in absorptance than seen for aging at lower temperatures. Furthermore, the rate of crack formation and the evolution of oxidization morphology may vary under cyclical testing conditions mimicking those experienced in real operating conditions due to cloud transients. This is an area of ongoing investigation and will be reported in further work.

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