



A novel non-selective coating material for solar thermal potential application formed by reaction between sol–gel titania and copper manganese spinel

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ABSTRACT

A method for preparing a novel bixbyite non-selective coating for solar thermal conversion is described. The coating is formed by a thermal reaction between a titania sol–gel precursor with a copper manganese spinel to form a new material, $\text{Cu}_{0.44}\text{Ti}_{0.44}\text{Mn}_{0.84}\text{Fe}_{0.28}\text{O}_3$, with a bixbyite structure. The effect of temperature and ratio between the two components on the formation of the bixbyite layer (deposited on Inconel by spray-coating) was studied. The absorptance of the films (AM 1.5; 335–2500 nm) with a thickness of $10 \pm 2 \mu\text{m}$ after annealing at 2 h at 650 °C and 750 °C was 97.4% and 94.7%, respectively. This synthesis represents a novel approach in which the final solar thermal coating is formed as a continuous and uniform layer which combines both the absorber and the ceramic binder. The developed material shows promising results for future applications as absorber in solar thermal energy conversion.

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1. Introduction

Solar thermal has become one of the leading approaches in solar energy conversion, which is used in concentrating solar power (CSP) systems. It is expected that by 2050, with appropriate support, CSP will provide more than 10% of global electricity. The common base of all CSP technologies and classifications is the solar absorber [1], which collects the solar spectrum and converts it into heat. These absorbers are subjected to extreme temperatures that vary between the operation temperature, which currently exceeds 500 °C, and < 0 °C at night. Moreover, it needs to meet certain requirements, such as corrosion resistance and severe abrasion by sand and dust.

Absorber surfaces are typically classified as selective and non-selective depending on the absorptance to emittance ratio. Whereas the selective coatings are mainly made by vacuum (physical and chemical) deposition [2] and sputtering [3,4] the non-selective absorber surfaces can be prepared also by wet chemistry. The latter are typically composed of a mixture of a black inorganic pigment, e.g. spinels and metal oxides, and a transparent matrix, usually an oxide, which serves as a binder. A

few excellent reviews on mid- and high-temperature absorber materials have been published over the years [5–7]. Surveying these studies reveals that only a few non-selective coatings that are made by wet chemistry processes are suitable for high-temperature applications. During the last years absorbers for higher temperature (> 500 °C) are sought as a means of improving the solar thermal conversion efficiency. One of the central reviews in this field [6] classifies mid- and high-temperature absorber materials to coatings operating at 100–400 °C and 400–700 °C, respectively.

Among the application techniques used for wet coating are roll- and coil-coating [8], electroplating [9] and spraying [10]. Most of the reported studies and patents are for mid-temperature applications. For example, a selective paint for mid-temperature was prepared from polysiloxane binder and an inorganic pigment, i.e. FeMnCuO_x , which was applied onto an aluminum substrate by a draw-bar coater followed by annealing at 300 °C [8]. Recently, polyurethane binder and black spinel pigment applied by either coil-coating on aluminum or spray-coating on copper showed good thermal stability at 200 °C and superior oxidation protection [10,11]. Electrodeposition was used to prepare black nickel coatings with absorptance (α)/emittance (ϵ) of 0.88–0.96/0.03–0.10 for temperature below 200 °C [9].

Very few studies reported high-temperature coatings for solar thermal applications made by wet deposition. For example, such a coating was prepared from polysilazane as a binder and Co_3O_4 as a

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spinel pigment on 304 stainless steel using spin-coating [12]. Spectrally selective absorber coatings were prepared by spin-coating of nickel nanoparticles embedded in alumina with an antireflective layer made of silica, alumina, or silica–titania mixture [13]. Tempil Pyromark[®] and Ergenics Novamet[®] are known commercial black paints for high temperatures [14]. In general using wet chemistry processes results in non-selective coatings as described by [5] for representative black paints or enamel coatings.

Hence, it is evident that the field of **high-temperature solar thermal coatings applied by wet chemistry** is still in its infancy and the development of such absorbers is of high relevance and importance in particular for renewing CSP systems, where coating must be carried out on-site. The ultimate goal for non-selective coatings is to obtain high absorptance (>95% of the solar spectrum) and uniform coating that is stable and durable at high temperature (>700 °C).

Here we report on a novel approach for obtaining a high absorptance coating for high-temperature based on the chemical reaction between titania sol–gel precursor and a spinel. We are aware of only a few studies where spinels were either incorporated in a titania matrix or doped with Ti [15–24]. For example, nanosized colored $Zn_xTi_yO_{w-z}N_z$ spinel was synthesized using sol–gel based ZnO nanoparticle precursors [18]. Macroporous Zn_2TiO_4 was formed by reacting TiO_2 nanoparticles milled with ZnO at air at 1200 °C [23]. Orel [24] reported doping of $CuCoMnO_x$ by Ti in a polysiloxane resin for mid-temperature (450 °C) solar thermal application. The films were used in order to increase the weather resistance and showed absorptance of 0.86–0.91 and emittance below 0.036.

In the present study we report on the thermal chemical reaction between a titania sol–gel precursor with the copper manganese spinel to form a new material, $Cu_{0.44}Ti_{0.44}Mn_{0.84}Fe_{0.28}O_3$, with a bixbyite structure. The effect of temperature and ratio between the two components on the formation of the bixbyite layer (deposited on Inconel by spray-coating) was studied. The absorptance of the films (AM 1.5; 335–2500 nm) with a thickness of $10 \pm 2 \mu m$ after annealing at 2 h at 650 and 750 °C was 97.4% and 94.7%, respectively. This synthesis represents a novel approach in which the final solar thermal coating is formed as a continuous and uniform layer which combines both the absorber and the ceramic binder.

2. Material and methods

2.1. Chemicals

Absolute ethanol, acetylacetone (acac, for analysis grade) and nitric acid were purchased from Merck. Tyzor[®] TE(D), (termed TE) was from Dorf Ketal (Mumbai, India) and ethyl cellulose (10 cP) was obtained from Sigma-Aldrich. Disperbyk-190 was received from Byk (Wesel, Germany) and Black 26 from Shepherd (Ohio, USA). Deionized water (EasyPure UV, Barnstead) was used for all experiments. Inconel 718 sheets were from Snappy Materials (CA, USA).

2.2. Instruments

Dispersions were prepared using a homogenizer (Polytron PT-2500E, Switzerland). The coating was applied by an air-brush (Paasche VL-SET double action siphon feed airbrush set). Thickness was evaluated by P-15 profilometer (KLA-Tencor Co., San Jose, CA, USA). Absorption measurements for the range of 300–2500 nm were carried out by SOC410-Solar visible/NIR hand-held reflectometer (Surface Optics Corporation, USA) and for the range of

360–740 nm by CM-2600d (Konica Minolta, Japan) reflectometer. Based on a modified integrating sphere, these instruments measure total reflectance at seven sub-bands in the 300–2500 nm spectral regions. Morphology and chemical analysis were performed with a SIRION scanning electron microscope (SEM, FEI Company) equipped with EDS detector. TEM imaging was performed by Tecnai F20 G² (FEI Company) equipped with an EDS detector (EDAX-TSL, USA). The oxidation states of the elements composing the tested material were studied by X-ray photoelectron spectroscopy (XPS, Kratos XPS Axis Ultra spectrometer). XRD measurements were performed with D8 Advance diffractometer (Bruker AXS, Karlsruhe, Germany) equipped with Göbel Mirror parallel-beam optics. XRD patterns from 5° to 95° 2θ were recorded at room temperature using $CuK\alpha$ radiation ($\lambda=0.15418$ nm) and step scan mode with a step size of 0.02° 2θ and counting time of 1 s per step for the preliminary study and of 12 s per step for structural refinement. The instrumental broadening was determined using LaB_6 powder (NIST-660a). TOPAS-v.3 software was used for structure refinement. Pseudo-Voigt function was used as a profile function for the Rietveld refinement.

2.3. Procedures

The coatings were prepared as follows: 40 mg of ethyl cellulose (EC) were dissolved in 6.1 ml EtOH with magnetic stirring. 0.5 ml deionized water and 20 μl of HNO_3 (11 M) were added, followed by addition of 2.2 ml acac and 5.0 ml TE. The molar ratio of the reactants TE:Acac:EtOH: HNO_3 :EC: H_2O in the coating mixture was 1:2.3:11.3:0.02:0.01:8.4. This mixture was left overnight with stirring. 413 mg of dispersant, Disperbyk 190, was added to the solution (in order to prepare 0.6 w/w of the pigment) followed by homogenization at 2,000 rpm for 1 min. 688 mg of the pigment Black 26 was then slowly added to the solution (yielding 1:3 w/w TiO_2 :Black 26) and homogenized at 7000 rpm for 10 min. Inconel sheets were rinsed with soap and water, followed by acetone and ethanol and finally dried prior to using. Coatings were applied by spraying a homogeneous layer on the Inconel sheets, then dried for 10 min at 50 °C and annealed by the following profile: 10 °C/min up to 80 °C, 30 min at 80 °C, 5 °C/min up to 350 °C, 30 min at 350 °C, 5 °C/min up to 750 °C and 2 h at 750 °C (or 650 °C in certain experiments). Samples for SEM inspection were manually ground and placed on a double-sided carbon tape glued to a standard SEM stub. TEM samples were ground manually in an Agat mortar. The powder was transferred onto a standard TEM nickel grid (400 mesh) by pressing.

3. Results and discussion

The overall goal of this study was initially to obtain a high absorptance coating that is stable at high temperature. This can be achieved by dispersing inorganic pigment particles within a ceramic matrix, the latter having good binding to the metallic substrate. Titania was selected as a binder and a commercial spinel pigment, Black 26, was used as the absorber. The optical properties of the coating will be presented first followed by material characterization. Interestingly, we found that a new material was formed by the thermal reaction between the inorganic pigment and the titania. This finding represents a new approach whereby the new material acts as both the absorber and the matrix.

3.1. Coating absorptance

The coating formulations were prepared by mixing the titania sol–gel precursor and Black 26 particles at 1:3 solid weight ratio, with the additives ethyl cellulose (rheological agent), acetyl

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