



Zirconia nanoparticles embedded spinel selective absorber coating for high performance in open atmospheric condition



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ABSTRACT

Spectrally selective absorber coatings which have high thermal stable at temperatures ≥ 500 °C in open air atmosphere are more beneficial for all types of concentrated solar thermal power (CSP) applications. In order to achieve this, high crystalline zirconia nanoparticles are synthesized via Lyothermal process were embedded into a spinel matrix to form a novel composite solar selective absorber layer (Mn-Cu-Co-O_x-ZrO₂). To enhance the absorption substantially, an ink-bottle type mesoporous MgF₂ nanoparticles are synthesized via novel route have been deposited further. As a result, a novel tandem absorber system (Mn-Cu-Co-O_x-ZrO₂/MgF₂) has been developed with $\alpha/\epsilon = 0.97/0.17$ @500 °C. The composite layer comprises of tetragonal phase Zirconia particles which were uniformly distributed in the spinel matrix responsible for high optical performance and high thermal stability. These properties are thoroughly investigated by micro X-ray diffraction (μ XRD), X-ray photoelectron spectrometer (XPS) and Elemental mapping by Energy filtered transmission electron microscope (EF-TEM) technique. This novel composite absorber layer developed by a facile route exhibits superior stability up to 700 °C ideally in the air makes a promising advancement for the cost-effective power generation by CSP systems.

1. Introduction

Concentrating solar thermal power (CSP) technology is capable of harnessing the immense solar resource and it is emerging as a key technology to generate electricity with a low level of greenhouse gas emission. In CSP, high-temperature can be obtained by concentrating the sun's rays with the aid of reflectors to produce electricity in a thermodynamic cycle. In CSP power plants the storage of thermal energy enables power generation even in the absence of solar radiation, which contributes to CSP's distinctive ability to provide dispatchable power [1,2].

Receiver efficiency and Carnot efficiency both play a major role in the overall efficiency of the concentrated solar thermal system. High photo-thermal conversion efficiency can be achieved with a spectrally selective coating (SSC) [3]. In order to get high Carnot efficiency, the temperature of heat transfer fluid (HTF) must be 600 °C or higher [4]. Employing SSCs which can be operated at 700 °C or higher can lead to a high temperature of HTF as well as Carnot efficiency. Therefore it is important for SSC's to possess high spectral selectivity (high absorptance (α_s) & low emissivity ϵ_T in the range of 0.3–2 μ m & 2–25 μ m respectively), high chemical stability, thermal stability and durability at an operating temperature or under regular variations in temperature.

Generally, High temperature solar selective absorber coatings can be obtained by Physical Vapor Deposition method, particularly sputtering [5–12], spark-erosion [13], cathodic arc PVD [14], atomic layer deposition [15] are applicable for medium and high-temperature applications. These methods lead to high costs due to large amount of material consumption, complex and expensive equipment and it could be a potential problem for cost effective production. Wet chemical methods such as Anodization [16–18], Electrochemical deposition [19–21] and chemical oxidation [22–24], have also been reported as low-cost methods in comparison to different vapor deposition techniques but coatings obtained by these methods do not offer high chemical and thermal stability. Moreover, methods like chrome electroplating, chemical oxidation are not environmental friendly.

From the material point of view, spinel materials have attracted great attention in the field of spectrally selective absorber coatings in the last decade because to their inherent high temperature and oxidation stability. In addition, Spinel are amenable to the substitution of a large number of transition metals, which can be chemically tailored to tune the optical properties. Many research groups have already attempted for the preparation of spinel structure using various materials for concentrated solar thermal energy conversion applications [25–29]. They faced a major challenge to obtain absorber layer with the

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combination of high solar absorptance and low thermal emittance with high-temperature stability in an open air atmosphere. The major drawback of using spinel structure materials as a solar selective absorber coating is that they have a high thermal emissivity at operating temperature.

Kaluzna et al., reported that a black colored CuFeMnO_4 spinel film deposited by sol-gel dip coating technique exhibited a satisfactory value of $\alpha = 0.85$ to 0.93 , but the surfaces show unsatisfactory thermal emissivity ($\varepsilon_T = 0.07$ – 0.62) [25]. By replacing Fe with Co, thermal emissivity was altered to $\varepsilon_T = 0.05$. However, it is not clear that if this layer keeps physical integrity at high temperatures [26]. Another research group reported incorporation of Ti into spinel structured material of CuCoMnO_x which intern was deposited on aluminum substrates. In spite of good optical properties ($\alpha > 0.85$ – 0.91 , and $\varepsilon < 0.036$ @RT) [27], there is no information on the stability of spinel layer. Layers made of transition metals like copper, cobalt, manganese and silicon mixed oxides form nanocrystalline films after annealing showed promising results. The solar absorptance and thermal emissivity of 0.95 & 0.12 respectively at 100°C was observed but limited by thermal stability up to 360°C [28]. Tulchinsky et al. reported the thermal chemical reaction between a Titania sol-gel precursors with the copper manganese spinel to form a new material, $\text{Cu}_{0.44}\text{Ti}_{0.44}\text{Mn}_{0.84}\text{Fe}_{0.28}\text{O}_3$, having a bixbyite structure. Although absorption of the film was reported around 97.4% , no measurement was done for thermal emittance and stability of the film [29].

Very few studies have reported good optical efficiency with high thermal stability for solar absorber coatings developed by incorporating suitable nanoparticles into composite absorber sol. For example, Xavier Paquez et al. [30] has reported a simple wet chemical route for a bilayer $\text{RuO}_2/\text{SiO}_2$ nanoparticles stack on a pre-oxidized stainless tube exhibiting thermal stability in the air up to 600°C with a solar selectivity ($\alpha > 0.94$, and $\varepsilon@600^\circ\text{C} < 0.28$). Nevertheless, there are still some challenges like high thermal emissivity and cost of the materials utilized for the absorber film.

Different from spinel materials, we previously demonstrated Mn-Fe-Cu composite layer developed on a special variety of Cu-rich austenitic stainless steel plate [31] by a controlled thermal oxidation process. This novel layer exhibits 0.91 of solar absorptance and 0.19 of emittance (@ 500°C) with high thermal stability up to 500°C in an open air atmosphere. The achieved properties were insufficient for commercial usage.

In this present communication, we reported the design and fabrication of a novel absorber system to attain high-performance absorber coatings which are thermally stable for temperature $\leq 700^\circ\text{C}$ in air with high spectral selectivity. Achieving high thermal stability by solution precursor method had been challenging task. However, we overcame it by incorporating Lyothermally synthesized crystalline nano zirconia particles into a spinel absorber solution (Mn-Cu-Co) and forming a composite nanoparticle layer. Absorption enhanced further with an aid of mesoporous MgF_2 nanoparticles layer on top of the absorber layer. Further, we reported the optical, structural and thermal stability along with preparation of single and tandem absorber layers which is essential for CSP application.

2. Experimental section

2.1. Synthesis of crystalline zirconia nanoparticles

The preparation of nanoparticle and their structural characterization are described elsewhere [32]. A clear solution of Zirconium precursor mixture was prepared with n-propanol, HCl and Deionized water by sequential mixing in the molar ratio of 25: 2: 1: 1 (n-propanol: Zr precursor: HCl: Deionized water). The mixture was stirred at room temperature until the clear suspension was obtained. Thereafter, the mixture was transferred to a Teflon coated autoclave and heated at 180°C for 3 h. After completion of the autoclave process, the nanoparticles present in the solution were agglomerated by adding non-

polar solvents preferably acetone and butanone for an easy separation from unused precursors and solvent mixture. Finally, the agglomerated nanoparticles were separated by centrifugation and dried under vacuum at 60°C for 4 h. The prepared dry nanopowder was dispersed in water and utilized for the preparation of homogeneous Mn-Cu-Co-ZrO₂ composite absorber coating sol.

2.2. Preparation of ink-bottle mesoporous MgF_2 nanoparticles

For the preparation of tandem absorber layer, high crystalline mesoporous MgF_2 nanoparticles were synthesized from micron size particles by lyothermal synthesis. The synthesis procedure of MgF_2 nanoparticles was exactly followed as mentioned in our earlier work [33]. It comprises of many steps, initially 10 g of magnesium fluoride hydrate ($\text{MgF}_2 \cdot \text{XH}_2\text{O}$ powder with average particle size of around $35\ \mu\text{m}$, Alfa Aesar) was mixed with 60 mL of n-propanol and 5 mL concentrated HCl (37%, Merck Millipore). A transparent solution was obtained by stirring the mixture at a constant rate at room temperature. Further, 2 mL deionized water was added to the mixture followed by mechanical stirring for an hour. Thereafter, the mixture was transferred into a Teflon coated autoclave and heated at 200°C for 1 h, to obtain high crystalline MgF_2 nanoparticles with uniform particle size, good weather and thermal stabilities. The prepared dry powder could be easily dispersed uniformly in polar solvents for forming the coating solution.

2.3. Synthesis of Mn-Cu-Co-ZrO₂ composite sol for preparation of Mn-Cu-Co-O_x-ZrO₂ nanocomposite film

Mn-Cu-Co-ZrO₂ composite absorber sol was synthesized by a combination of impregnation and sol-gel methods at room temperature. The hydrated salts such as manganese acetate, copper chloride, cobalt chloride were used to synthesize Mn-Cu-Co absorber sol. All these chemicals were purchased from E. Merck and used without further purification. A stable solution of Mn-Cu-Co composite absorber solution was obtained after dissolving these optimized amounts of precursors successively in alcohol preferably ethanol. The weight ratio between manganese, copper and cobalt was optimized as 4.6:3.2:1.5 respectively in order to obtain best optical and thermal stability. Thereafter, the nano zirconia suspension was added dropwise into Mn-Cu-Co composite sol. Finally, the mixture was stirred and aged for 24 h at room temperature.

2.4. Preparation of MgF_2 nanoparticle suspension for preparation of tandem absorber layer

The mesoporous MgF_2 nanoparticles were finely dispersed in a 90:10 mixture of ethanol and isopropoxy ethanol (Alfa Aesar) to obtain a stable suspension. The prepared sol was used to obtain a uniform thin film on Mn-Cu-Co-ZrO₂ composite nanoparticle layer using the dip-coating method. To obtain maximum optical properties of the tandem absorber layer, the MgF_2 layer speed was varied between 2 mm/sec Section – 4 mm/sec. It was noted that the film showed a high solar absorptance and low thermal emissivity at an optimum withdrawal speed of 4 mm/sec.

2.5. Substrate preparation and deposition of absorber & tandem layers

The polished tube of austenitic stainless steel (304) was used as a substrate and its typical size was 50 mm length, 1" OD and 1.5 mm thickness. The samples were cleaned by ultrasonication for 15 min, washed with water and a soap solution. After that, samples were rinsed with tap water followed by de-ionized water and finally wiped with soft cotton with a solvent to make substrate free from oil and grease to get uniform coating and better adherence. Subsequently, substrates were dried in hot air oven at 100°C for 10 min. Following the cleaning procedure, the film deposition was carried out on a substrate in a closed

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