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## Black oxide nanoparticles as durable solar absorbing material for high-temperature concentrating solar power system



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### ABSTRACT

Concentrating solar power is becoming an increasingly important part of the renewable energy portfolio. However, further cost reduction is desired to make CSP competitive with traditional energy technologies. Higher operating temperature is considered an attractive avenue leading to higher power conversion efficiency and lower cost, but tremendous technical challenges exist with higher temperature operation of CSP, with one of the main issues being the lack of a high-performance solar absorbing material that is durable at 750 °C or above. In this work, a black oxide material, made of cobalt oxide nanoparticles, is synthesized and utilized as a high-temperature solar absorbing material. The nanoparticles are embedded in a dielectric matrix through a scalable spray coating process. The top layer of the coating is further improved with light-trapping structures using sacrificial fillers introduced from the same coating process. After the surface modification of cobalt oxide coating, we achieved a high thermal efficiency of 88.2%. More importantly, the coating shows no degradation after 1000-h annealing at 750 °C in air, while the existing commercial light absorbing coating was reported to degrade by long-term exposure at high temperature. Our findings suggest that the materials and processes developed here are promising for solar absorbing coating for future high-temperature CSP systems.

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### 1. Introduction

The development and deployment of renewable energy sources is becoming an increasingly urgent need for human society [1,2]. Concentrating solar power (CSP) systems (or solar thermal systems) are becoming an important part of the major portfolio of renewable energy generation. One of the key potential advantages of CSP over many other forms of the renewables is the possibility of inexpensive energy storage using thermal energy storage systems (TES), which is useful for grid-level power management [3]. The TES can extend the electricity generation capability to periods with no sunlight available, thereby significantly expanding the value and usage of solar energy [4]. CSP systems can also be hybridized with other alternative energy systems, such as solar photovoltaic [5], thermoelectric [6,7], or thermophotovoltaic [8,9] systems, to increase the penetration of renewable energy power [10,11]. Despite these perceived benefits, the leveled cost of

energy (LCOE) of CSP, however, is still too high to compete with traditional thermal power plants and some other alternative energy technologies (such as photovoltaics) [12].

In order to develop a cost-competitive CSP technology, it is imperative to increase the system power conversion efficiency. To maximize the efficiency of CSP, it is desirable to raise the operating temperature for higher Carnot efficiency. As a result, the temperature of heat transfer fluids (HTFs) needs to be 700 °C or higher [13]. Toward this aim, all of the components of CSP systems, such as solar field, HTFs, power block, TESs, and solar receivers need to be made compatible with the higher-temperature operation.

As far as the solar receiver is concerned, the light-absorbing coatings on the receiver play an important role by absorbing solar thermal energy and raising the temperature of the HTFs to above 700 °C. Although spectrally selective coatings (SSCs) with multi-layers/graded cermet or tandem structures have been developed and stably operated in vacuum in parabolic trough CSP systems [14–19], none of these SSC structures can operate at high temperatures in air, which is needed in future solar-tower based CSP systems. The state-of-the-art solar absorbing material used in solar towers is based on a commercially available black paint called Pyromark 2500<sup>®</sup>. Pyromark 2500<sup>®</sup> is known to have high thermal efficiency above 600 °C and has been used for central solar receivers

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of CSP plants. However, according to prior studies, the absorptivity degraded after high temperature ( $> 700\text{ }^{\circ}\text{C}$ ) exposure due to crystal structure changes and phase instability [20,21]. Therefore it is clear that a new light absorbing coating material with high absorption efficiency and excellent durability at elevated temperature in air environment needs to be developed for the next generation CSP systems.

Since the light absorption is directly related to solar energy generation efficiency, a large number of studies about optical absorption enhancement have been actively pursued. Several light trapping approaches were studied, such as texturing [22–25], metallic nanostructures [26], and photonic [27–29] and plasmonic structures [30,31]. However, most of these studies about light trapping have been applied on photovoltaic applications using vacuum deposition methods. As a result, these processes are not necessarily compatible with CSP applications, where spray coating is generally employed to coat the light absorbing layers [21,32] onto solar tower receivers, for example, Pyromark 2500<sup>®</sup> in Solar One and Solar Two central towers [33,34].

Here, we report a highly efficient and high-temperature durable light absorbing coatings based on cobalt oxide nanoparticles for CSP receivers. Cobalt oxide materials have been studied as selective absorbing layers and fabricated using spray pyrolysis method [35–37], chemical vapor deposition [38] and electrodeposition. Although several previous studies showed the applicability as solar collectors within the temperature range of  $300\text{--}650\text{ }^{\circ}\text{C}$  [39–41], there is no existing light absorbing coating suitable for CSP operating at higher temperature ( $\sim 750\text{ }^{\circ}\text{C}$ ) without performance degradation. In this study, the cobalt oxide nanoparticles were synthesized via a facile hydrothermal process and utilized as the light-absorbing material in the coating layers. The coating layers consist of cobalt oxide nanopowders dispersed in silica matrix, and are deposited on metal substrates via a simple and scalable spray coating process, which is compatible with CSP applications. We employed novel and yet simple surface texturing techniques, based on sacrificial polymer beads that can be easily integrated with the spray coating process, to improve the light absorption. Finally, the developed coating layer exhibited unprecedented high-temperature durability, showing no degradation in structural or optical properties after annealing at  $750\text{ }^{\circ}\text{C}$  in air for 1000 h.

## 2. Experimental

### 2.1. Cobalt oxide nanoparticle synthesis and sample preparation

We first aimed at synthesizing cobalt oxide nanoparticles of right sizes for high light absorption. For nanoparticles (aspect ratio  $\sim 1$ ), the optimal size for absorbing visible and near infrared light is probably around  $200\text{--}400\text{ nm}$  [42]. If the particle size was too small, the resultant coating film would have small surface roughness, which is not effective for light trapping. In addition, for high temperature application, very small nanoparticles ( $< 100\text{ nm}$ ) would agglomerate and become larger size particles. Therefore, we aimed at producing nanoparticles with hundreds of nanometers in diameter. Metal oxide nanoparticles can be prepared by a number of different methods, such as sol–gel technique [43], mechanical grinding (ball milling) [44], mechanochemical synthesis [45], and hydrothermal synthesis [46], etc. Here we utilized the hydrothermal method to synthesize  $\text{Co}_3\text{O}_4$  nanoparticles for convenience. However, it should be noted that other inexpensive nanoparticle synthesis techniques mentioned above may also be used for producing metal oxides for SSC applications.

Cobalt oxide nanoparticles were synthesized via a hydrothermal process using cobalt chloride salt ( $\text{CoCl}_2\cdot 6\text{H}_2\text{O}$ ) as the precursor.  $10\text{ M}$

solution of sodium hydroxide ( $\text{NaOH}$ ) was gradually dropped into  $1\text{ M}$  solution of cobalt chloride to induce precipitation of cobalt hydroxide until pH value of the reacted solution reached 11. The hydrothermal synthesis was performed at  $150\text{ }^{\circ}\text{C}$  for 20 h in order to transform the precipitated cobalt hydroxide into cobalt oxide. The cobalt oxide particles were then washed with de-ionized water using a centrifuge and dried using a freeze-dryer. The as-synthesized nanoparticles were annealed at  $750\text{ }^{\circ}\text{C}$  for 2 h for phase stabilization.

For the fabrication of  $\text{Co}_3\text{O}_4$  slurry, methyl phenyl polysiloxane resin (SILIKOPHEN<sup>®</sup> P 80/X) was utilized as precursor of  $\text{SiO}_2$  matrix. The volume ratio between the active material (cobalt oxide nanopowders) and the  $\text{SiO}_2$  matrix was systematically varied and optimized. The required amount of resin was diluted with an organic solvent mixture consisting of two miscible solvents, xylene and isobutanol (volume ratio 3:1). The optimum dilution was important to ensure good quality of the coating layers, because too viscous slurry could not make a smooth spray stream while very dilute slurry could not make a stable spray-coating onto substrates. In order to dissolve the resin in the solvent, the mixture was sonicated for 30 min with a probe type sonicator. During sonication, the solution container was cooled in an ice-bath to avoid concentration change due to the evaporation of organic solvents.  $\text{Co}_3\text{O}_4$  powders were mixed with the prepared solution and yttria stabilized zirconia (YSZ) grinding balls were also added into the blend. The ball milling was conducted for 24 h in order to make the mixture homogeneous.

The black oxide spray coating was performed using a spray gun on a high temperature Ni alloy (Inconel 625) sheet coupons ( $\frac{1}{2}\text{ in} \times \frac{1}{2}\text{ in}$  in size). The spray pressure was set at 40 psi and the distance between the spray gun and the Inconel substrate is about 10 cm, which has been optimized for the best coating quality. After spray coating, the samples were heated at  $250\text{ }^{\circ}\text{C}$  for 1 h for curing of  $\text{SiO}_2$  resin. All of the coating samples consist of two layers: the base layer of approximately  $30\text{ }\mu\text{m}$  thick and a top layer with surface-topography-modified structure described in the next paragraph.

### 2.2. Cobalt oxide surface texturing

The first method to alter the surface texturing and topography of the top layer to improve optical absorption was to employ imprinting stamps with SU-8 polymer pillars which were prepared by using standard microfabrication processes. The polymer pillars were  $3\text{ }\mu\text{m}$  in both diameter and spacing and  $10\text{ }\mu\text{m}$  in height. The stamps were then pressed onto coated  $\text{Co}_3\text{O}_4$  coating surface and left as imprinted prior to the resin curing step. Then, the sample was annealed at  $750\text{ }^{\circ}\text{C}$  for 1 h to burn away the remaining polymer pillars.

The second method employed to create roughened surface is by incorporating and subsequently removing micron-sized sacrificial polymeric beads within the  $\text{Co}_3\text{O}_4$  coating layer. The volumetric concentration of  $\text{Co}_3\text{O}_4$ , polymer beads and silicone resin was optimized to yield the best optical performance (as shown in Table 1), and the mixtures were again sonicated with a probe type sonicator for proper mixing. Then, a desired amount of silicone resin was added, followed by the same ball milling and spray coating processes described in the prior paragraphs. Finally, the

**Table 1**  
Sample fabrication condition and figure of merits (FOM).

Sample name	Volume ratio			FOM (figure of merits)
	$\text{Co}_3\text{O}_4$ nanopowders	$\text{SiO}_2$ dielectric matrix	Polystyrene polymer beads	
$\text{Co}_3\text{O}_4$ -1	1	1.5	0	<b>0.854</b>
$\text{Co}_3\text{O}_4$ -2	1	4.5	3	<b>0.877</b>
$\text{Co}_3\text{O}_4$ -3	1	6.5	5	<b>0.882</b>

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