



Delineating the role of surface characteristics on the solar selectivity of colored chromium oxide coating on 304 stainless steel substrate



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ABSTRACT

Solar selective colored chromium oxide coatings were prepared on the SS304 substrate through the electrochemical deposition route to provide more options and flexibility in aesthetically design solar absorbers with acceptable energy performance. The variation of colors in the coatings was achieved by varying the coating thickness as a function of deposition time. Color changes in the coatings were also evident in the samples that were annealed under vacuum. Electron microscopic images show that nanometer sized pores are present on the coating surfaces. The pore-density, pore-size distribution and surface roughness exhibit significant changes with variations in deposition time and annealing temperature. Initially, the selectivity factor of 6.08 was recorded in the as prepared sample coated for 30 min, which improved to 7.36 on annealing the samples under vacuum at 700 °C, however, it drops to 4.22 when annealed at 900 °C. A similar trend was also observed for samples that are coated for 60 min. The results indicate that the pore-density, pore-size distribution and surface roughness play a dominant role in governing the solar selectivity of the coatings. Nanoindentation tests were performed on the coated samples and the results show that mechanical properties of the solar absorber coatings degrades with the increase in coating thickness but improve significantly with increase in annealing temperature.

1. Introduction

The search for solar selective absorber material is becoming increasingly prominent with the growing demand of solar energy harvesting. Solar selective absorbers play an important role in all major solar thermal technologies e.g. concentrated solar power (CSP), solar thermo-electric generators (STEG), and solar thermo-photovoltaic's (STPV) for harvesting energy from the sun [1–3]. The most important criteria for the solar absorber material are to absorb sunlight without losing heat to the environment. In this regard an ideal solar selective absorber should have very high solar absorptance and low infrared (IR) emittance properties [4–6]. The past decade has witnessed an extensive research on developing ideal solar absorber material in the form of thin film coatings to attain the desirable solar selectivity by employing different coating techniques (e.g. electrochemical, sputtering, spray coating, spray pyrolysis, chemical vapour deposition, electroplating, anodizing, sol-gel, spin coating, dip coating etc.) [7]. Among these various solar absorber materials, metal-dielectric composites and cermet(s) received special attention due to their high solar absorptance, low IR emittance and good thermal stability [7,8].

Till date the most widely used and commercially available cermet

absorber material is black chromium oxide (Cr_2O_3) based cermets which possesses the ability to trap and expand the optical path of the solar energy, which induces a high solar absorptance in it [7,9,10]. Though it is evident that black surfaces deliver the highest solar absorptance, they are not always considered aesthetically acceptable in all cases, e.g. facade integration [11–13]. During the past few years, interest on the development of colorful solar selective absorber is seen with the objective of expanding the usage of solar technologies with a colorful architectural integration. Many studies have been reported on colored solar absorber using multilayer structures of various metal-dielectric composites [8,14–16], but availability of reports on the research work focused on the development of colorful Cr_2O_3 based absorber are limited and more importantly detailed study on the thermal stability, mechanical property and the effect of surface morphology and microstructure of the colored Cr_2O_3 coatings on the solar thermal property of the material is yet to be reported. Considering the importance of the Cr_2O_3 based colored absorber, the present study attempt to electrochemically develop a colored Cr_2O_3 based cermets coating over the stainless steel substrate. The electrochemical deposition route was selected due to its low complexity, high control over the process parameters, low cost, good reproducibility and possibility of scale up.

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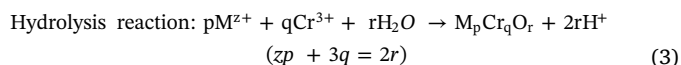
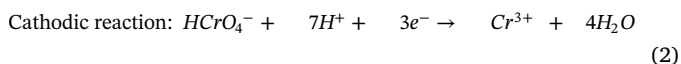
On the other hand the high thermal conductivity, excellent corrosion resistance property and the low emissivity are the reasons behind the selection of stainless steel as substrate. The mechanical properties of the coating were determined by using nanoindentation technique. Studies on the thermal characteristics and stability of the coatings were carried out at elevated temperature and the effects of the surface morphology and microstructure on the solar thermal property was also investigated

2. Experimental

2.1. Development of coating

A commercial grade SS304 sheet of thickness 75 μm , with the weight percent composition; Cr- 18.36, Ni- 8.06, Mn- 0.93, Si- 0.49, S- 0.01, C- 0.06, P-0.04, Mo-0.36, Cu-0.43, N-0.08 and balance Fe was used as the substrate. For colorization, 30 mm x 62 mm size samples were cut from the SS304 sheet and each of them were ultrasonically degreased for 10 min in acetone followed by rinsing in distilled water prior to the colorization treatment. The cleaned samples were colored electrochemically by immersion in a mixture of hot (70 °C) aqueous 5 mol/L H_2SO_4 and 2.5 mol/L CrO_3 solution under a constant current density of 3.8 mA/cm². The electrochemical bath consists of SS304 sheet (cathode), a reference platinum electrode (anode), a DC power supply and a heating mantle to raise the temperature of the bath to 70 °C.

During the colorization treatment, an oxide film was formed on the SS304 substrate by dissolution and precipitation mechanism according to the following reaction sequences [17],



where, M represents the alloying elements present in the SS304 sample viz. Fe, Cr, Ni, Mn etc. and M^{z+} is the corresponding ions formed by anodic dissolution reaction, viz. Fe^{3+} , Cr^{3+} , Ni^{2+} , Mn^{3+} . The cathodic reduction of chromic acid yields Cr^{3+} ions and finally, a heavily hydrated oxide coating was formed by the hydrolysis reaction between the metal ions produced by dissolution of the SS304 steel and Cr^{3+} ions formed by reduction of chromic acid, leading to precipitation of the oxide layer on the substrate. The thickness of the coating was varied by varying the coating time for 30 min and 60 min. The stability and characteristics of the coating surfaces with respect to elevated temperatures were studied after annealing the coated samples in vacuum (5×10^{-5} Torr) at 500 °C, 700 °C and 900 °C for 1 h. The sample designations with respect to the processed conditions are shown in Table 1.

2.2. Characterization

The microstructure and surface morphology of the coated films were studied with the help of field emission scanning electron microscope, FESEM (JSM-7610F, JEOL, Japan). The thickness of the coating, pore

Table 1
Sample designation with respect to their processing conditions.

Sample specification	Sample identity	
	30 min deposition	60 min deposition
As coated	C30	C60
500 °C annealed	500C30	500C60
700 °C annealed	700C30	700C60
900 °C annealed	900C30	900C60

size and their distributions were measured from the FESEM images recorded from different locations of the sample surface. Atomic force microscope, AFM (Dimension Edge, Bruker, Germany) was used to measure the roughness of the coating surfaces. The AFM images were acquired in the peak force tapping mode, using a silicon nitride tip at room temperature. The roughness values were evaluated after image processing of $100 \times 100 \mu\text{m}^2$ scan areas from several locations and are expressed as the root mean square roughness (R_q). The coefficient of surface skewness (S_q) and kurtosis (S_k) was also determined from the AFM images. All AFM image processing work was carried out with the help of Nanoscope analysis software package (Version 1.4). Thermogravimetric and differential thermal analysis were performed on the as coated samples under static air medium with the aid of a thermal analyzer (TG-DTA, model Diamond, Perkin Elmer, US) in the temperature range of 30–900 °C at a heating rate of 10 °C/min. The mechanical properties of coatings were investigated with the help of nanoindentation workstation (NHTX 55-0019, CSM instruments, USA) equipped with a berkovich diamond indenter. All nanoindentation tests were performed under a maximum load of 1 mN with 10 s of dwell time. The low load was selected to avoid the influence of the substrate. For each sample, a minimum of 10 (e.g. 5×2 array) indents were taken at randomly selected locations on the coating surfaces. The results were averaged and presented with the standard deviation.

The thermo-optical properties viz., solar absorptance (α_s) of the samples were determined by a solar spectrum reflectometer (SSR-E, Devices and Services Co., USA) for the entire solar region (200–2500 nm) as per ASTM C1549-09. An average hemispherical IR-emissivity (ϵ_{IR}) of the samples were also measured by an emissometer (AE, Devices and Services Co., USA) at infrared (IR) region (3–30 μm) as per ASTM C1371-04a [18].

3. Result and discussion

3.1. Effect of coating thickness

The surface of the coated samples was scratched with a sharp razor blade and the scratched section was studied under the FESEM for determining the thickness of the coating. Fig. 1 shows the representative FESEM images of the samples with the coating thickness and the results are presented in Table 2. The results indicate that the coating thickness of the samples increases with the increase in deposition time. A conspicuous change in color from dark blue to lime gold was also observed with the increase in coating thickness, which can be ascribed to the variations in the refractive index of the coating promoted by the changes in the coating thickness. Annealing the samples at 500 °C causes marginal change in the color and thickness of the coating surfaces, because of the simultaneous evaporation of physically attached water and reaction between unreacted metal surfaces with the residual acidic solution which remained trapped in the pores of the oxide coating. However, the change in color and reduction of thickness becomes very prominent when the samples were annealed at higher temperature. The reduction of coating thickness at temperatures above 500 °C is mainly instigated by the elimination of lattice bound water molecules, trapped acidic solutions in the pores and may also be caused by the collapse of pore walls. The corresponding FESEM images show that no distinct interface boundary exists, indicating an excellent bonding between the coating and the substrate, which continues to exist even after annealing at 900 °C.

3.2. Surface morphology

The representative microstructural features of the coated surfaces of the samples are shown in Fig. 2. Presence of nanopores on the coated surfaces is observed in all samples. Formation of nanopores arises because, during the electrochemical deposition process, when the Cr^{3+} concentration at the metal/solution interface exceeds the solubility

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