The effect of annealing temperature on selective solar absorptance of Ni-Al coating prepared by flame spray technique

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Abstract

Ni-Al coating has been considered a promising candidate as a solar absorber material in solar thermal applications operating at mid-to high-temperatures. In a previous study by the current authors, a Ni-Al composite layer was applied as a coating onto a stainless steel 316L pipe using the flame spray technique. The solar absorptance ($\alpha_{\text{sol}}$) achieved, however, was relatively low compared to other commercial solar absorber coatings. To enhance the $\alpha_{\text{sol}}$ of the Ni-Al, the annealing process was performed on this coating. The results clearly revealed that the metal oxides on the surface of the Ni-Al coating were NiO and Al$_2$O$_3$. As the annealing temperature increased, the reflectance ($R$) slightly decreased, while the $\alpha_{\text{sol}}$ significantly increased. This was caused by the layer of metal oxides covering the surface of the Ni-Al coating functioning as an antireflection (AR) layer. The $\alpha_{\text{sol}}$ of the Ni-Al coating increased approximately 13% after annealing at 800°C, compared to the unannealed Ni-Al coating. It is therefore clear that the annealed Ni-Al coating enhances the $\alpha_{\text{sol}}$ due to the antireflection effect between the metal oxide layer and the underlying Ni-Al composite coating.

Keywords: Selective absorber; Annealing; Ni-Al, Absorptance; Solar absorber; Antireflection
1. Introduction

Fossil fuels (coal, oil, gas) have long been used as sources of energy, and are known to contribute to pollution and climate change. However, more recently, have seen growth in renewable energy, which are investigating potential ways to move the world with green energy. Among the available renewable energy sources, solar energy is considered to have great potential due to being more reliable, inexhaustible, non-polluting, abundant, available everywhere as a free energy source [1, 2]. Solar collector and concentrated solar power (CSP), using photo-thermal conversion, is one of the major established technologies which can transfer solar radiation directly into a usable form of heat [3]. Solar collectors can be classified, based on the temperature operation range, as: a) low temperature (<100°C) b) mid-temperature (100°C<T<400°C) and c) high temperature (>400°C) [1, 4]. In Photo-thermal conversion systems, solar absorbers are important components for trapping and converting solar radiation into thermal energy. In order to achieve the maximum efficient photo-thermal conversion applications, spectrally selective solar absorbers have been designed to absorb more solar energy with less heat radiation loss. Theoretically, a selective solar absorber requires a high solar absorptance ($\alpha_{\text{sol}}$) as much as possible in the UV/VIS/NIR region of the solar spectrum (300-2500 nm) and a low thermal emittance ($\varepsilon_T$) or high reflectance ($R$) in longer wavelength infrared (IR) region (2.5-25 μm) [3-7]. Researchers are attempting to develop the selective solar absorbers which achieve the best spectral properties, high-temperature stability, and low cost. A transparent ceramic anti-reflection (AR) layer is usually coated on top of the solar absorber coating layer in order to obstruct the reflection of solar radiation from a solar absorber surface, as a result of the reflective index mismatch between air and the solar absorber layer [8]. As with previous reports, AR materials include metal oxides which are used to improve $\alpha_{\text{sol}}$ and reduce $\varepsilon_T$ of the selective solar absorber in a suitable wavelength range such as Al$_2$O$_3$ [1, 6], SnO$_2$ [6, 9, 10], SiO$_2$ [2, 10-12], TiO$_2$ [2], AlSiO$_y$ [7], AlCrO$_x$ [3], and SiON [5]. In addition, AR coating layers have been prepared using various techniques such as the sol-gel method [8], the dip coating technique [11], DC/RF sputtering [2, 5-7, 10], the cathodic arc ion plating system [3], and heat treatment [13].

Ni-Al coatings have been extensively developed for a solar absorber of CSP receiver applications in mid- to high operating temperature ranges [13, 14]. Ni-5Al coating on 304L stainless steel prepared by air plasma spray (APS) torch exhibited $\alpha_{\text{sol}}$ and $\varepsilon_T$ (80°C) to be 0.63 and 0.39, respectively, and it enhanced the $\alpha_{\text{sol}}$ to be 0.89 by heat treatments for 6 hours at 600°C [13]. In a previous study, Ni-Al coating was successfully prepared on a stainless steel 316L pipe using the flame spray technique which obtained $\alpha_{\text{sol}}$ of 0.77 over the solar spectrum range [14]. A further improvement to achieve high $\alpha_{\text{sol}}$ and low $\varepsilon_T$, an AR layer is required on the top Ni-Al coating.

In this study, therefore, the AR layer on top Ni-Al coating was prepared using the annealing method. The effect of annealing temperature on phase, microstructure, chemical composition, reflectance and solar absorptance is investigated and discussed.

2. Experiment

Ni-Al coating on 316L stainless pipe was prepared using the flame spray technique with a commercial starting material of Ni-5wt.%Al particles (Metco 450NS, Oerlikon, Switzerland). The details of the flame spray gun, equipment setup, and fuel conditions were previously proposed [14]. The samples were provided by cutting the Ni-Al coating with the length 10 mm and width 10 mm. The various annealing temperatures were 400, 600, and 800°C with a ramp rate of 5°C/min. The Ni-Al coating samples were annealed in air atmosphere for 6 hours in a furnace. After annealing, the samples were gradually cooled down to room temperature under the natural condition of furnace.

The phase and crystallinity of the samples were analyzed by X-ray diffraction (X’Pert MPD, Philips). To investigate the effect of annealing temperature on the cross-sectional microstructure, and chemical composition of the Ni-Al coatings, the samples were characterized by using a Scanning electron microscope (SEM, JEOL JSM-5910LV) equipped with an energy-dispersive X-ray (EDX) analyzer. The reflectance ($R$) of the samples were measured using a Shimazuzu UV-3101PC spectrophotometer with a wavelength of 300-2500 nm, which corresponds to the wavelength of the solar spectrum range. The normal solar absorptance ($\alpha_{\text{sol}}$), is theoretically defined as a weighted ratio of the absorbed radiation to incoming solar radiation, was calculated using measured spectral
reflectance data, \( R(\lambda) \), and the solar spectral irradiance, \( I_s(\lambda) \), at air mass AM 1.5 (ASTM G173-03), as shown in Equation [3, 7, 8]:

\[
\alpha_{sol} = \frac{\int_{0.3 \mu m}^{2.5 \mu m} I_s(\lambda)(1 - R(\lambda))d\lambda}{\int_{0.3 \mu m}^{2.5 \mu m} I_s(\lambda)d\lambda}
\]  

(1)

3. Results and discussion

All XRD patterns (Fig. 1) of the Ni-Al coatings were indexed and compared with JCPDS database of Ni, NiO, Al, and Al\(_2\)O\(_3\) with reference nos 4-0850, 1-1239, 1-85-1327, and 47-1292, respectively. It is observed that all samples composed of Ni, NiO and Al\(_2\)O\(_3\) phases. The Al phase was not found in all coatings because it rapidly reacts with oxygen gas in the air, to form an Al\(_2\)O\(_3\) phase during the flame spray process and annealing. The intensity of diffraction peaks of NiO and Al\(_2\)O\(_3\) phases increased with increasing the annealing temperature, while the Ni phase decreased. As the annealing temperature increased, the intensity of NiO (200) and Al\(_2\)O\(_3\) (220) peaks sharply increased. The Ni-Al coating annealed at 800°C. The intensity of NiO (200) peak was more than any other Ni peaks. According to a previous study, the results indicated that the Ni and Al elements on the surface of the Ni-Al coating were formed to NiO and Al\(_2\)O\(_3\) phases during the annealing process [13]. Upon characterization of the Ni-Al coating before and after annealing at different temperatures, the Ni-Al coating annealed at 800°C presented the highest NiO and Al\(_2\)O\(_3\) phases covering the underlying Ni-Al composite layer, compared with the other samples. Furthermore, NiAl, AlNi\(_3\) or Al\(_3\)Ni\(_5\) were not found in the Ni-Al coating after annealing, as results of the no-driving reaction between Al and Ni elements due to the low Al content [15].

![Fig. 1. XRD patterns of Ni-Al coating for before and after annealing at different temperatures.](image-url)
To further ascertain the presence of metal oxides (NiO, Al₂O₃) covering the underlying Ni-Al composite layer, the samples were characterized using SEM. Fig. 2 shows the cross-section SEM images of microstructure Ni-Al coating with different annealing temperatures. It is observed that the Ni-Al composite coating was built up to overlap the melted Ni and Al particles layer by layer. The result of the melted Ni and Al particles injected from the flame spray gun at high velocity and acceleration to create a top surface layer with a somewhat rough surface. There are three different contrast regions: light gray, gray and dark gray. The thickness average of Ni-Al coatings were identical (~170 µm). The microstructure of Ni-Al coatings corresponded to the related Ni-Al compounds prepared with several techniques [16-18]. According to the un-melted and imperfectly melted particles of starting material during the flame spraying process, the pores were formed with different sizes in the Ni-Al coatings [14]. It was found that a different contrast layer was found on top Ni-Al coating after annealing for 6 hours at 800°C (Fig. 2c), compared with other annealing temperatures. Fig. 2d shows the Ni-Al coating after annealing for 6 hours at 800°C. It is indicates that both metal oxides of NiO and Al₂O₃ formed on the surface of the Ni-Al coating which, according to the XRD results high NiO and Al₂O₃ phases. To a layer of metal oxides, oxygen in the air was able to sufficiently penetrate and distribute into the matrix of the Ni-Al composite layer during annealing at 800°C. The thickness of the metal oxide layer depended on the surface morphology and type of metal elements.

Fig. 2. Cross-section SEM images of Ni-Al coating at different annealing temperatures; (a) 400°C, (b) 600°C and (c, d) 800°C.

Fig. 3. Ni-Al coatings before and after annealing: (a) Measured R and calculated αsol spectra and (b) the αsol over the whole range of the solar spectrum.
The measured $R$ and calculated $\alpha_{sol}$ spectra of the Ni-Al coatings before and after annealing at different temperatures were compared in Fig. 3a, together with the sun spectrum at AM 1.5 (ASTM G173-03). There is some change in the $R$ between pristine and annealed coatings. The $R$ in the solar spectrum region of all samples tended to increase with increasing wavelength. With increasing annealing temperature, the $R$ of the Ni-Al coatings significantly decreased. The best $R$ spectrum in the whole range of the solar spectrum was Ni-Al coating after annealing at 800°C, due to increasing metal oxides to form a layer and complete the crystallization process with the stoichiometric composition of NiO and Al$_2$O$_3$ phases on the top layer of the coating (Fig. 1-2) [10]. The reasons could be the effect of the metal oxides covering the Ni-Al coated surface after annealing, which leads to a reduction in the reflective index mismatch between air and the Ni-Al coating. It is indicated that the layer of metal oxides was the AR layer caused by a lower reflective index than the Ni-Al composite layer [3, 11]. As the results of the $\alpha_{sol}$ spectra versus wavelength (Fig. 3a), $\alpha_{sol}$ of Ni-Al coatings presented high values at 300-800 nm and tended to decrease with the increasing wavelength, corresponding to the results of the $R$ spectra. In additions, the $\alpha_{sol}$ of the coatings increased with the increasing annealing temperature due to the effect of the AR layer on trapping the solar irradiance. Fig. 3b presents the $\alpha_{sol}$ of Ni-Al coatings over the whole wavelength of the solar spectrum. The $\alpha_{sol}$ gradually increased with the increasing annealed temperature and reached a maximum value of 0.85 after annealing at 800°C, according to the $\alpha_{sol}$ spectra in Fig. 3a. The $\alpha_{sol}$ of the Ni-Al coating increased about 0.1 or 13% after annealing at 800°C, compared to unannealed Ni-Al coating. The coating exhibited good thermal stability in air atmosphere at 800°C for 6 hours. Furthermore, the maximum $\alpha_{sol}$ of Ni-Al coating annealed at 800°C ($\alpha_{sol} = 0.85$) closed to the Ni-5Al solar absorber prepared by air plasma spray (APS) torch after heat-treated for 6 hours at 600°C ($\alpha_{sol} = 0.89$) [4], and solar absorber materials for CSP technology for mid-to high-operating temperatures [1, 12]. It is, therefore, clear that the Ni-Al coating on 316L stainless pipe prepared by the flame spray technique is able to enhance the $\alpha_{sol}$ by annealing, and presenting good thermal stability.

4. Conclusions

In summary, the $\alpha_{sol}$ of Ni-Al coatings on 316L stainless pipe prepared by the flame spray technique were successfully enhanced by annealing at different temperatures. The coatings composed of Ni, NiO and Al$_2$O$_3$ phases. The intensity of diffraction peaks of NiO and Al$_2$O$_3$ phases increased with increasing the annealing temperature, while Ni phase decreased, indicating that the Ni and Al elements on the surface of Ni-Al coating were formed to NiO and Al$_2$O$_3$ phases during annealing. As the results of SEM images, A layer of metal oxides covering the top Ni-Al coating after annealing at 800°C were found to be the NiO and Al$_2$O$_3$, while the layer of metal oxides was not found in the pristine and annealed Ni-Al coating at 400°C and 600°C. With increasing annealing temperature, the $R$ of the Ni-Al coatings significantly decreased, while the $\alpha_{sol}$ of the coatings gradually increased. These reasons could be the layer of metal oxides as the AR layer, leading to suppress the reflective index mismatch between air and the Ni-Al coating caused by the lower reflective index than the Ni-Al composite layer. The $\alpha_{sol}$ of Ni-Al coatings reached a maximum value of 0.85 after annealing at 800°C, indicating a 13% enhancement of $\alpha_{sol}$ when compared to the $\alpha_{sol}$ value without annealing. It can be concluded that the Ni-Al coating on the 316L stainless pipe prepared by the flame spray technique is a potential candidate of solar absorber material in the solar collectors operating at mid-to high-temperatures.

Acknowledgements

The authors would like to acknowledge the National Research Council of Thailand (NRCT), and Naresuan University, Thailand for providing financial support (Grant no. R2560B113). Thanks also to the NULC Writing Clinic for editing the manuscript.

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