



Influence of nanoporous aluminum oxide interlayer on the optical absorptance of black electroless nickel–phosphorus coating

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ABSTRACT

This paper introduces a technique to make an ultra-black surface by employing nanoporous anodized aluminum oxide as a template and deposition of nickel–phosphorus nanowires by the electroless process. The optical properties were compared with two other processes; a conventional black Ni–P deposition and a nickel electro-coloring process, on aluminum substrate. Surface morphologies of the samples were examined by field emission scanning electron microscope and elemental analysis of the coatings was performed by the energy dispersive spectroscopy method. Optical properties of surfaces were determined by spectrophotometry and infrared spectroscopy techniques. In addition, optical characteristics of the coated surfaces were evaluated by calculation of absorption and emission coefficients of the surfaces. The results showed that ultra-black duplex coating possessed an absorption coefficient higher than 99%, while emission coefficient decreased about 6% compared with simple black electroless Ni–P. Calculation of ξ factor indicated that a value of 5.1 proved that optical properties in the duplex coated sample had a significant improvement.

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1. Introduction

Solar energy in the form of heat absorbed by a black surface can be used in photo thermal systems. The main part of the collector utilized for the production of solar energy is the absorber and the needed black absorber surface can be produced by deposition of an appropriate film on a metal substrate [1]. The absorber may be selective or non-selective; selective solar absorber coatings form the basis of a wide range of high performance optical coatings and interference filters. A selective surface is achieved by applying a thin coating of high absorptivity on a metal surface of low emissivity, obtained by several techniques [2]. Since aluminum has light weight, low cost, good thermal conductivity and low emittance, it has been used extensively as a perfect substrate for optical coatings [3,4].

Anodizing, a conventional process to improve surface properties of aluminum alloys, has been used to produce black surfaces [5]. Since the porous structure of anodic oxide layer formed on the surface of aluminum is used as a template to make such structures as nanowires and nanotubes, it could be considered as an interlayer for impregnation of various pigments [6]. A black anodized surface may be produced by electrolytic coloring techniques. In this process, metal particles are deposited into the pores of the anodic oxide coating during the half cycle

of alternating current. Coloring of aluminum oxide may be achieved by organic dyes or by chemical or electrochemical deposition of inorganic compounds inside the pores.

Various black anodizing processes such as inorganic dyes, electrolytic coloring, precipitation pigmentation, or combinations of organic dyeing and electrolytic coloring have been studied for optical applications [7,8]. In these processes, several elements including nickel, copper and tin are used as pigments; due to high absorption, low cost and ease of manufacture, nickel layers have been used more than other coatings.

Electrolytic coloring pigmented aluminum oxide solar absorber was first suggested in 1979 [9]. Although it has a good optical performance, the absorber is sensitive to abrasion. Electroless plating is an appropriate technique to deposit nickel layer, regardless of geometry of the substrate, ideal for a wide range of applications in metal finishing industries. Nickel electroless coating has a combination of properties such as corrosion resistance, hardness and lubricity [10]. Electroless plating of nickel is an autocatalytic process for deposition of metal coatings on certain catalytically active substrates using a controlled chemical reaction [11].

Many selective solar absorber designs are possible, but here the selection must be focused on a composite coating over a metallic substrate of sufficiently high infrared (IR) reflectance. In this research, an electroless deposition was applied onto anodized surface of aluminum substrate in order to construct a duplex layer. The duplex coating was compared with electro-colored anodizing and electroless Ni–P in terms of optical properties. Surface properties and optical characteristics of the coatings were evaluated and absorption coefficients of the coatings were studied. The optimum black surface was introduced in

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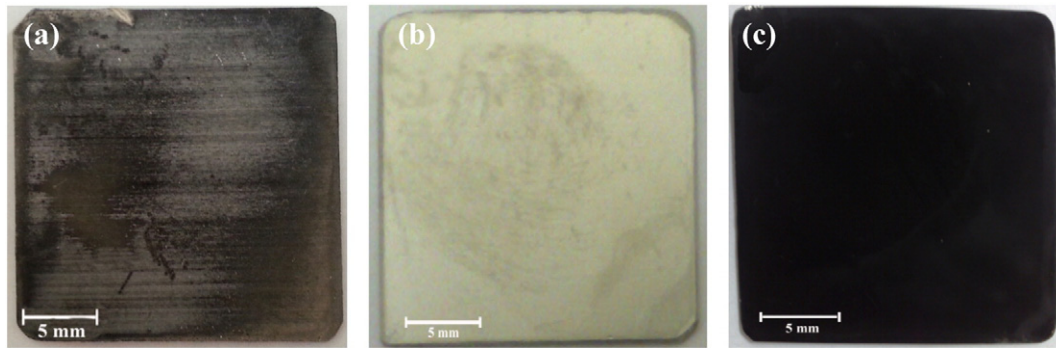


Fig. 1. Duplex coatings of different interlayer thicknesses; (a) very thin, (b) thick, (c) optimized.

terms of ξ factor; an appropriate absorber should be selective to possess high solar absorptance (α) and low thermal emittance (ϵ).

2. Experimental details

2.1. Formation of coatings

Commercially pure aluminum sheets (Al-1100) were cut into $20 \times 20 \times 1$ mm samples and cleaned ultrasonically in acetone bath for 5 min. The samples were degreased using 10% sodium hydroxide at $60\text{--}70^\circ\text{C}$ for 5 min, rinsed and immersed for 1 min in nitric acid at room temperature. Anodizing process was carried out in an electrolyte containing 165 g/L H_2SO_4 . A range of anodization times and dc voltages were selected in order to prepare coatings of different thicknesses. The temperature was in the range 13 to 15°C . Thickness of the porous anodic oxide films was determined by an eddy current thickness meter model Salu Tron®D2.

To produce ultra-black surfaces, three methods were applied; first, conventional electroless Ni–P coating was applied directly on aluminum substrate in a commercial Schloetter solution; SLOTONIP 70A with a pH of 5 at $85\text{--}90^\circ\text{C}$ and 750 rpm agitation speed. The deposition rate in this solution was between 18 and $22\text{ }\mu\text{m/h}$. After deposition, blackening process was performed by immersion of samples in 9 M nitric acid solution at 40°C for 20 s. In the second method, electroless Ni–P film was applied over an anodized interlayer to achieve the required surface. Finally, the third set of samples was prepared by electrolytic coloring applied onto anodized interlayer for 10 min using ac voltage of 15 V in nickel bath with a pH of 5.6 at 25°C ; the electro-coloring bath contained 37 g/L $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$, 22 g/L $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$, 65 g/L $(\text{NH}_4)_2\text{SO}_4$ and 26 g/L H_3BO_3 with stainless steel 316 L used as a counter electrode at a constant current of 0.45 A/dm^2 . In the two latter processes containing interlayers, the thickness of anodized aluminum oxide (AAO) was optimized based on the quality of black coatings.

2.2. Evaluation of coatings

Coating morphologies were characterized by scanning electron microscope (SEM), Philips model XL30, as well as field emission scanning electron microscope (FESEM), Hitachi model S4160. Energy dispersive spectroscopy (EDS) analysis was performed by INCA Oxford Instrument for determination of chemical composition of the coatings; incident electron beam energy of 21 keV was selected at a take-off angle of 35° and a measurement time of 50 s. The phases present in the coatings were analyzed by X-ray diffraction using Philips Expert-MPD equipped with monochromator at 40 kV, 30 mA and CuK α radiation ($\lambda = 1.542\text{ \AA}$). Measurement configuration was Bragg–Brentano ($\theta\text{--}2\theta$) geometry and the scan step was 0.5° per second. Optical properties of the surfaces, including absorption, emission and ξ factor were evaluated from the results of spectrophotometry (Jasco model V-570). The radiation reflected by the samples was measured in the wavelength range $360\text{--}1700\text{ nm}$. Fourier

transform infrared spectroscopy (FTIR) was performed by a spectrometer model 680 PLUS Jasco for determination of emission of the coatings; the radiation reflected by the samples was measured in the wavelengths $2500\text{--}25,000\text{ nm}$.

3. Results and discussion

3.1. Anodized coating

A range of voltages and time intervals were used to prepare several anodized films on the aluminum substrate as interlayer. In the electroless deposition of these samples, some of the anodized surfaces failed to give a uniform black surface; some thicker layers quickly changed to a nickel appearance surface with remarkable fine cauliflower morphology as observed by SEM. These samples did not produce a black

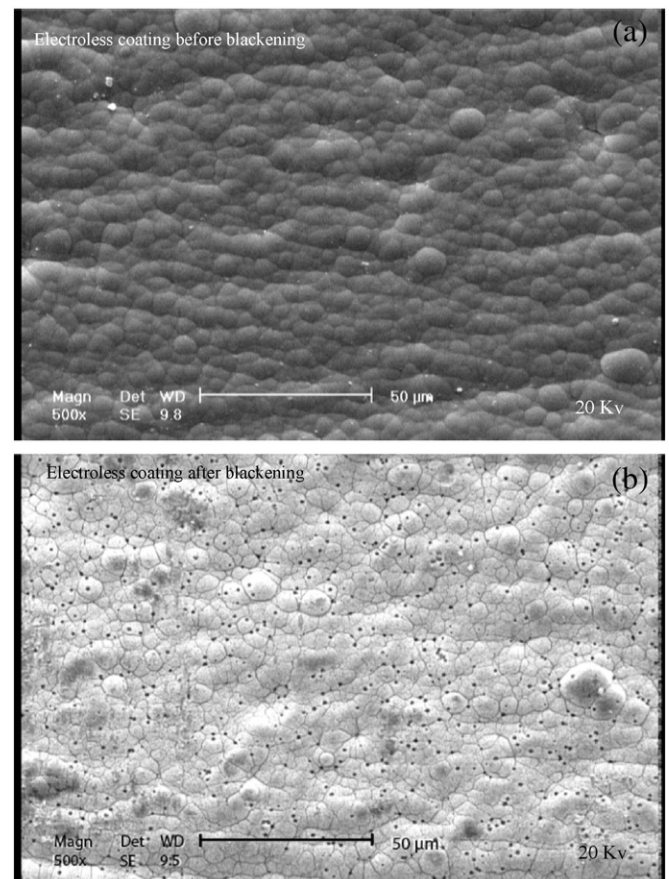


Fig. 2. FESEM micrographs of the electroless coating before and after blackening.

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