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Characterization of as-deposited and annealed Cr–Zn–P coatings electrodeposited from a trivalent chromium bath

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Black Cr–Zn–P coatings were electrodeposited on a steel substrate from a trivalent chromium bath using glycine as a complexing agent, ZnO as a second main component and sodium dihydro-phosphate as a source of phosphorus. Scanning electron microscopy (SEM), X-ray diffractometry (XRD), X-ray photoelectron spectroscopy (XPS) and spectral reflectance in the UV–visible–near IR and medium IR ranges were used to characterize the black coating before and after annealing. The relationship between the microstructure and optical properties of the black Cr–Zn–P coating deposited on the steel substrate has been studied using as-deposited and annealed samples. The deposits were found to have nodular crystals of various sizes with microcracks undergoing changes during heat treatment. XRD patterns suggest that the Cr–Zn–P alloy is a solid solution, which undergoes a series of transformations during annealing at 600 °C. The XPS results suggest that the near-surface layer is rich in Cr and Zn hydroxides, whereas oxides become dominant towards the substrate/coating interface. Besides, small amounts of phosphor and organic substances were also present in the coating. The phase constitution of black chromium varies by heating the sample up to 600 °C. The black Cr–Zn–P coating has good optical properties and these properties remain practically constant even when heat treated to a high temperature of 400 °C. It has been found that sample annealing at 600 °C initiated an increase in reflectance caused by the changes in the structure and chemical composition of the deposit and the diffusion of the substrate material (Fe) into black chromium.

Keywords: black Cr–Zn–P coating, morphology, optical properties, annealing

INTRODUCTION

Black chromium is widely used in solar collectors due to its high absorption, good stability and high thermal resistance. Traditionally, the solar selective coatings are deposited on metallic substrates. An effective selective coating is characterized by high absorptivity in the solar radiation range of 0.3 μm $<\lambda<$2.5 μm and stability at high temperatures. In order to improve optical characteristics of the deposits, more attempts have been made to obtain black alloy coatings as new materials for functional application. Black deposits containing chromium are used under the name "black chromium" in solar collector manufacture. For the selective coating optimization, it is essential to clarify the relationship between the chemical composition of black chromium and its thermal degradation. It is known that co-depositing of nickel and cobalt with chromium improves thermal resistance of black chromium [1]. Black Cr–Zn coatings deposited from the Cr(III)+ZnO bath were found to have good optical properties [2]. However, the absorptivity of such coatings decreases substantially after annealing at 600 °C [3]. According to the published data, the optical degradation of black chromium during annealing may be conditioned by oxidation of metallic chromium particles and diffusion of the substrate material into the coating. The black Cr–P coating is characterized by good optical properties with high solar absorption ($\alpha \approx 0.90$) [4]. According to the XRD patterns the as-prepared Cr–P deposit is of crystalline nature with uniform distribution of P and Cr in the alloy [5]. The occurrence of new phases at elevated temperatures is related to the recrystallization of the coating as a result of which the increase in grain sizes takes place. In order to improve the colour and optical properties of black chromium, some additive materials may be used [6]. It is obvious that the optimized chemical composition of the bath

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and optimum plating conditions are responsible for the highest absorptivity of black chromium.

Our interest in electrodeposition of the black Cr–Zn–P coating resides in the search for a new material suitable for the use in solar thermal systems. With this purpose sodium dihydro-phosphate was used as an additive to the Cr(III)+ZnO bath.

EXPERIMENTAL

Black Cr–Zn–P coatings were electrodeposited on the steel substrate, which was mechanically polished with further electropolishing before plating in accordance with the method described before [2]. The current density during plating was 0.2–0.4 A · cm^{-2} at a temperature of 20 °C. The analytical grade chemicals (manufacturer AppliChem GMBH) and distilled water were used to prepare the plating bath. The optimum bath composition and plating parameters for good quality of the deposit are presented in Table 1. The black coating was assessed visually and by using an optical microscope. A deposit of good appearance and good adhesion was defined as appropriate for further research. The coatings were heat treated at 400 and 600 °C for 2 h in air atmosphere.

Table 1. **The base composition of the black Cr(III) bath**

Component concentration, g dm^{-3}
$CrCl_3$ $6H_2O$ 250
NH_2CH_2COOH 18.75
NaCl 60.0
$NaNO_3$ 2.5
NaH_2PO_4 $2H_2O$ 6.0
ZnO 5.0
pH = 1.2
Temperature, 20 °C
i_c = 0.2–0.3 A cm^{-2}

The surface of black chromium coatings was examined by scanning electron microscopy (SEM). In order to choose a typical picture for each coating, three to four locations (in the central zone) were taken.

The phase structure of black chromium was characterized by the X-ray diffraction method (XRD) using an X-ray diffractometer Smart Lab (Rigaku) equipped with a 9 kW rotating Cu anode X-ray tube. The grazing incidence (GIXRD) method was used in the 2θ range 30–75°. The angle between the parallel beam of X-rays and the specimen surface (ω angle) was adjusted to 0.5°.

Elemental analysis of the coatings and the valence state of elements were studied by XPS. The spectra were recorded with a Vacuum Generator (VG) ESCALAB MK II spectrometer. Non-monochromatic Al K_α X-ray radiation ($h\nu$ = 1486.6 eV) was used for excitation. The Al twin anode was powered at 14 kV and 20 mA. The photoelectron take-off angle was 45° with respect to the sample surface normal and spectra of Cr2p, Zn2p, P2p, O1s, C1s and N1s were taken at the constant analyzer energy mode (20 eV pass energy). The base pressure was kept below 5×10^{-8} Torr in the working chamber. The spectrometer was calibrated in reference to Ag3d5/2 at 368.0 ± 0.1 eV and Au4f7/2 at 83.8 ± 0.1 eV. XPS depth profiling was performed in the preparation chamber where the argon gas pressure was maintained at 6×10^{-5} Torr with 50 liters/sec pumping speed at the gauge. Every time after sputtering the specimen was carried immediately to the analyzer chamber to avoid the rest gas adsorption. The quantitative elemental analysis was performed by determining peak areas and taking into account empirical sensitivity factors for each element [7, 8]. A standard program was used for data processing (XPS spectra were treated by Shirley-type background subtraction and fitted with mixed Gaussian–Lorentzian functions).

Spectral reflectance measurements were used to investigate the optical selectivity of samples coated with black chromium. Relative diffuse and specular reflection spectra were measured in a Shimadzu UV-3600 two beam spectrometer equipped with a multi-purpose compartment MPC-3100 by means of an integrating sphere of the 60 mm diameter coated by barium sulphate. Also the barium sulphate target was used as a diffuse reflective standard.

RESULTS AND DISCUSSION

It was found experimentally that the black coatings of good quality may be deposited from the bath presented in Table 1 over the current density range 0.2 to 0.3 A cm^{-2}. As the current density increases to 0.4 A cm^{-2}, the degradation of both colour and homogeneity of the deposit takes place. It is well known that the physical properties of electrodeposited coatings depend on their structural characteristics. The grain size of the deposit was analyzed by scanning electron microscopy (SEM). The influence of the current density on the surface morphology of black chromium is shown in Fig. 1a, b. The deposit obtained at a lower current density (0.2 A cm^{-2}) shows a loose structure made up of nodular crystals of various sizes: from 84 to 160 nm in diameter, among which the particles of 110 nm are predominant. There is a small number of globular particles (about 250 nm in diameter), which are agglomerations of small- and medium-sized particles. At a higher current density (0.3 A cm^{-2}), the surface looks more glossy with distinct nodular crystals surrounded by microcracks. The emergence of microcracks is related to intensive hydrogen evolution at higher current densities. The coatings suitable for solar thermal applications should have enough resistance toward temperature elevation. Therefore, black chromium samples were annealed at 400 and 600 °C (Fig. 2a–d). It is seen that after annealing at a temperature of 600 °C the deposit became rougher with wider and dipper cracks compared to the sample annealed at 400 °C.

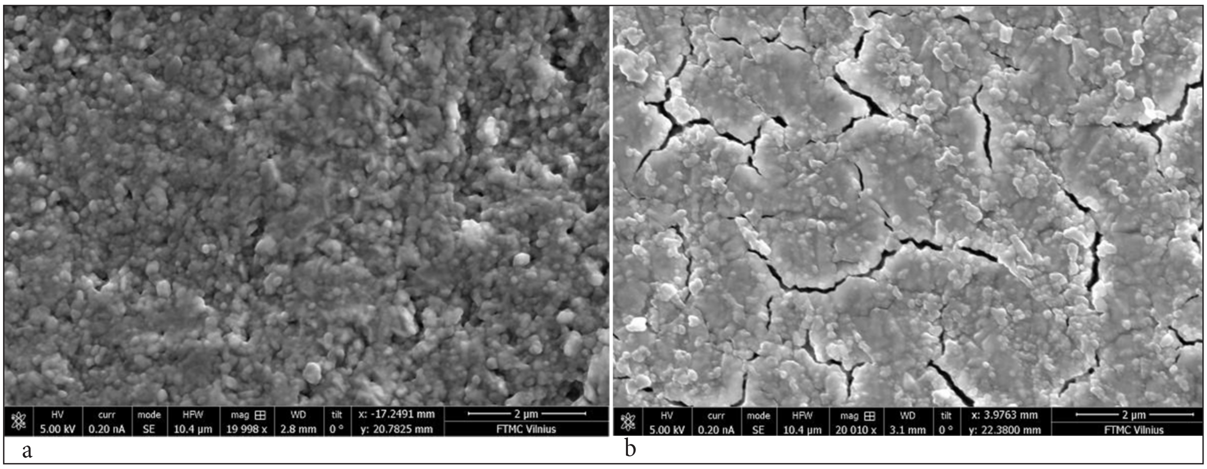

Fig. 1. Surface morphology of the black Cr–Zn–P coatings as-deposited on steel substrate at the current densities 0.2 A cm^{-2} (a) and 0.3 A cm^{-2} (b)

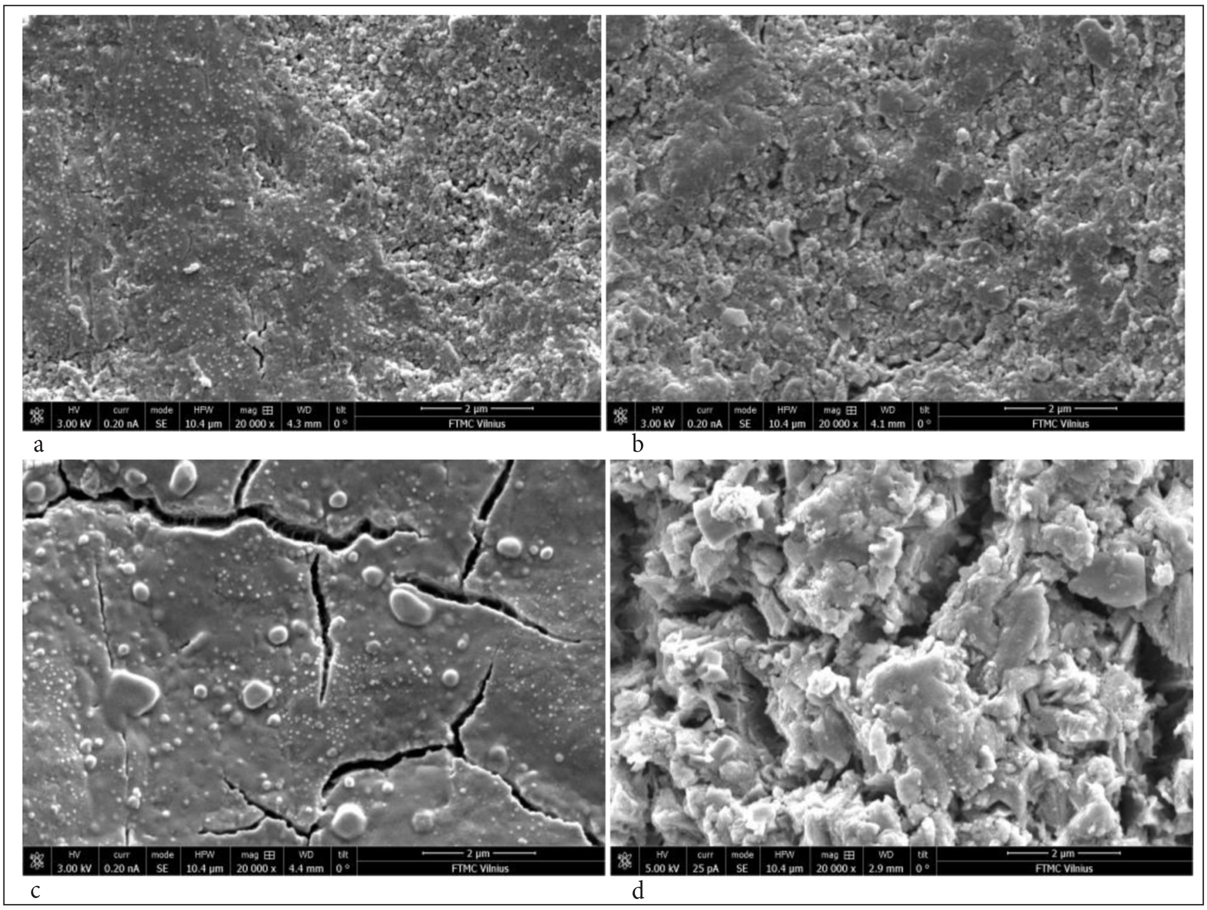

Fig. 2. Surface morphology of the black Cr–Zn–P coatings after annealing of samples at 400 °C (a, c) and 600 °C (b, d). The coatings were deposited at 0.2 A cm^{-2} (a, b) and 0.3 A cm^{-2} (c, d)

The deposit crystallographic structure was determined by the XRD method. In order to analyze the deposit structure, X-ray diffraction patterns were compared with JCPDS cards. Figure 3b shows an XRD pattern of the as-deposited Zn–Cr–P alloy. It is seen that there are sharp and broad peaks, which may be assigned to Zn and Cr structures, and a scarcely visible peak of the steel substrate. Because a sharp peak of zinc is too high (the scale of y-axis is confined to 1000 intensity (cps)), to reveal such a metalloid as P was impossible on account of its negligible quantity in the alloy. Again, it should be taken into account that the presence of metalloids such as P and C leads to the formation of amorphous structures. The peaks, which are attributable to the hexagonal structure, show lattice parameters a = 0.272209 nm and

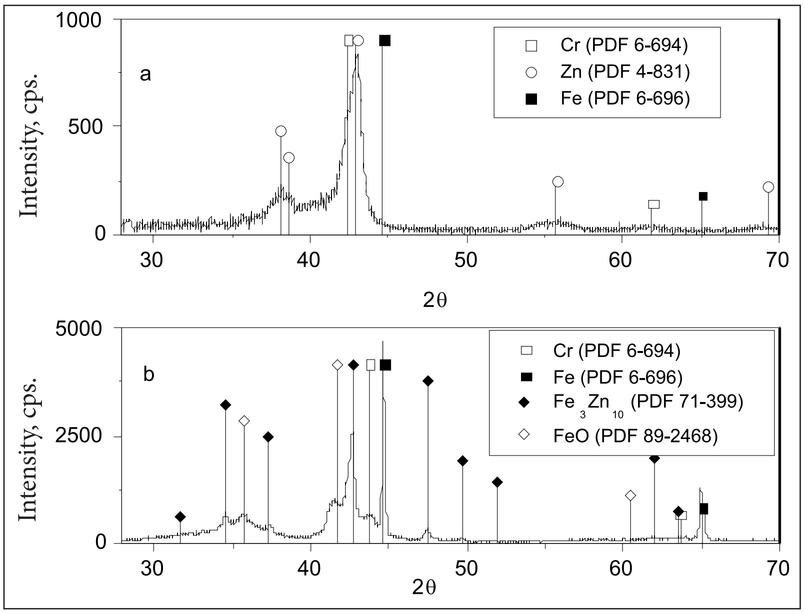

Fig. 3. XRD patterns of Cr–Zn–P coating before (a) and after annealing at 600 °C (b)

c = 0.466549 nm, which somewhat differ from those of metallic zinc a = 0.2665 and c = 0.4947 nm (according to ICDD data base card # 00-004-0831). The lattice parameter (*bcc*) of Cr equals 0.299794 nm instead of 0.28839 nm (# 00-006-0694). This implies that the chromium solid solution in zinc of hexagonal structure (*hcp*) was formed along with that of zinc in chromium (*bcc*). It was found experimentally that the thermal stability test at 400 °C did not cause the appearance of any new characteristic features in the XRD pattern, which suggests that the samples under study were stable at temperatures up to 400 °C. However, after sample annealing at a temperature of 600 °C a few additional peaks emerged in the XRD pattern (Fig. 3a). The sharp peaks at 2θ about 44.7° and 65.0° indicate that there exists a substance of a crystal structure dictated by the steel substrate (α-Fe) due to microcracks. Two less sharp peaks at 2θ about 35.8 and 42.0° are assigned to the cubic structure of FeO (a = 0.2920 nm), which is a result of oxidation of the steel substrate surface during the annealing process. The peaks at 2θ about 34.5; 37.5; 43.0; 47.5° correspond to the Fe_3Zn_{10} alloy (*bcc*) structure (a = 0.8988 nm) resulted from a series of transformation of the solid solution during annealing at 600 °C. After annealing of the sample, the lattice parameter (*bcc*) of Cr was found to be reduced (a = 0.2920 nm) owing to partial failure of the zinc solid solution in chromium.

To analyze the composition of black chromium, XPS analysis was performed. Table 2 lists the movement of the binding energy (BE) of the Cr2p3, Zn2p3, P2p, C1s and O1s peaks in the unresolved spectra from surface to depth and the atomic concentrations of components in the top layers of black chromium. To determine the quantitative compositions of the layers, it was necessary to develop an XPS deconvolution analysis. The chemical states of elements were identified by comparison of the photoelectron binding energies (BE) obtained with those reported in literature [7, 8]. After sputtering of top layers (45–120 nm), the Cr2p3 spectra were deconvoluted into three components with the dominant peak at BE = 576.5 ± 0.2 eV, which may be attributed to chromium oxides (Cr_2O_3, CrO_x) and double oxide ($ZnCr_2O_4$) [9]. Two modest-sized peaks

Table 2. **XPS depth profile of black Cr–Zn–P coating recorded before and after annealing at 600 °C for 2 h**

Element	Before annealing									
	Surface BE at %		6 nm BE at %		15 nm BE at %		45 nm BE at %		120 nm BE at %	
Cr2p3	576.06	0.16	577.01	0.72	576.94	1.15	576.79	3.76	576.80	7.58
Zn2p3	1022.3	0.10	1022.27	0.27	1022.16	0.53	1022.23	1.72	1022.08	4.00
P2p	134.80	0.43	133.85	0.60	133.79	0.62	133.21	0.55	134.39	0.53
O1s	532.26	12.41	530.95	8.94	530.57	12.66	530.69	14.94	530.46	36.24
C1s	284.66	85.53	285.01	85.83	285.02	79.44	284.80	72.36	284.83	53.77
After annealing										
Cr2p3	576.88	14.92	576.78	19.65	576.67	21.24	576.75	22.75	576.75	24.58
Zn2p3	1021.6	10.02	1021.82	8.92	1021.74	10.20	1021.71	9.49	1021.84	9.70
P2p	133.64	2.29	134.14	1.82	134.14	1.64	134.39	1.46	133.30	1.43
O1s	530.84	62.30	530.65	60.72	530.42	62.63	530.56	59.54	530.49	63.51
C1s	284.81	8.79	285.12	6.70	284.94	3.19	284.86	2.39	285.29	2.92

in the deconvoluted Cr2p3 spectra suggest the presence of $Cr(OH)_3$ (BE = 577.4 eV) and the metal-organic compounds (BE ~580 eV) which dominate on the surface, though a few of them were detected even at a depth of 120 nm. It is known [10] that inorganic species in Cr(III) complexes may be replaced by the organic ligands (L) forming various organo-chromium complexes, which may be captured by the growing deposit. These results are supported by the deconvoluted O1s spectra, among which the peak (BE = 530.4 ± 0.2 eV), corresponding to the metal–oxygen bonds in the oxide compounds, dominates. The other peak (BE = 531.6 ± 0.6 eV), which also was present in all depths, may be associated with the OH^- group from the coordination environment of Cr(III) and Zn(II). The position of a high energy peak (BE = 532.8 ± 0.4 eV) is associated with oxygen in organic fragments which, once involved in the coordination surrounding of Cr(III) ions, may be captured by the growing deposit. Therefore, a negligible high-energy peak was recognized even at a depth of about 120 nm in the deconvoluted Cr2p3 spectrum. The results suggest that the near-surface layer is rich in hydroxides, whereas oxides become dominant towards the substrate/coating interface. It was found that after annealing of the sample the major component in the deconvoluted Cr2p3 spectra shifted to a lower BE value in both outer and inner layers, which suggested that oxides became dominant from surface to depth of the coating. Besides, the emergence of the peaks at BE = 579.3 eV and BE = 574.3 eV in both surface and depth layers points to the presence of CrO_3 and metallic Cr phases in the annealed sample. The high energy peak (BE = 533.8 ± 0.4 eV) which is associated with oxygen in organic fragments, was not detected after annealing of the sample.

As for Zn2p3 spectra, it is not easy to characterize the individual components, because according to the literature data [8], BE of metallic Zn^0 (BE = 1021.6 eV) coincides very closely with that of Zn(II) compounds (BE = 1021.2–1022.0). The $Zn2p_{3/2}$ peaks at BE = 1022.1–1022.7 eV may be associated with $ZnCr_2O_4$ and $Zn(OH)_2$, whereas the peak at BE = 1021.4–1021.9 eV points to the presence of $ZnFe_2O_4$ and/or ZnO [8]. The peak at BE = 1021 eV, which emerges in the deconvoluted spectra after sample annealing, can be due to the metallic Zn bonds formed via heating.

The data in Table 2 show that after annealing the total carbon content decreases dramatically.

Analysis of the deconvoluted C1s spectra made it possible to reveal several components, which suggested the presence of distinct carbon bonds. Figure 4 shows that the C1s spectrum recorded on the surface of as-deposited black chromium consists of five components. It is generally believed that the peak at BE > 285.0 eV points to the presence of specific functional organic groups, whereas a low energy peak (BE < 285.0 eV) is associated with carbon–metal interaction and points to the presence of coordination bonds between the carbon chains and chromium atoms. The value of a negative shift depends on the electronic density on carbon. The low-energy component (BE = 283.8–284.6 eV), which emerged in the deconvoluted C1s spectrum may be assigned to the hydrocarbon fragment and the coordination bond between the carbon chains and chromium atoms [11]. The high-energy components, which may be assigned to C-N and C = N bonds (BE of 286.2 eV) and to C-H and C-C bonds (BE = 288.7 eV) in the organic compounds, testify that organic substances are involved into the black chromium deposit and its amount significantly decreases in depth. These components were found to be absent in the annealed sample. The emergence of the low-energy component with BE = 283.2 ± 0.2 eV after annealing suggests that a negligible amount of Cr_7C_3 may be formed during annealing at 600 °C. The reason is probably that such compounds as Cr(III) hydroxides and carboxylates, which were encapsulated in the deposits, transformed into Cr_7C_3 and oxides after heat treatment.

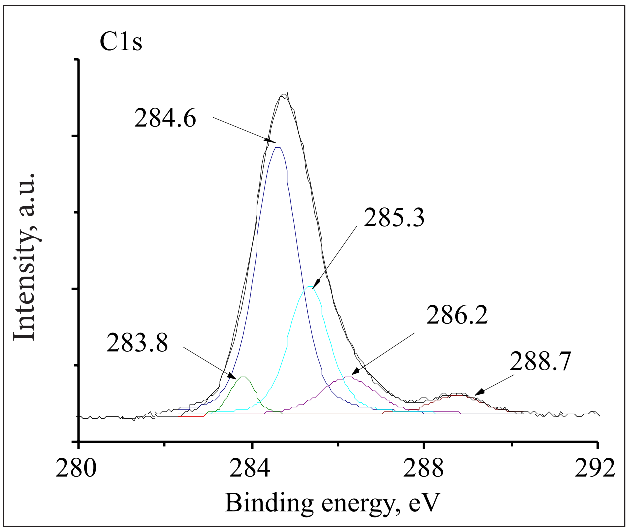

Fig. 4. XPS spectrum of C1s band recorded on the surface of as-deposited Cr–Zn–P coating

It is seen from Table 2 that the O1s peak position shifts towards lower binding energies into the depth of the coating, which suggests that the organic compounds and hydroxides dominate on the surface of the as-deposited coating. Analysis of the deconvoluted O1s spectra made it possible to elucidate the chemical state of atoms. The peak at BE in the range 529.9 to 530.6 eV, which is assigned to oxygen in the oxide compounds (Cr_2O_3 and ZnO), and the peak at BE = 531.6 ± 0.6 eV, which is associated with the OH^- group from the coordination environment of Cr(III), were detected in all layers. The position of high energy peak (BE = 533.8 ± 0.4 eV) is associated with oxygen in organic fragments which, once involved in the coordination surrounding of Cr(III) ions, may be captured by the growing deposit, therefore, it was detected even at a depth of about 120 nm. However, this component was not detected after sample annealing. The results obtained suggest that after annealing the oxides become dominant from surface to depth. This finding is in line with that of the above-considered Cr2p3 spectra.

As for the P2p band, the XPS spectra of the deposited alloy show a peak at BE = 133.8 ± 0.4 eV, which corresponds to the positively charged state and can be attributed to P^{5+} species. A weak nitrogen signal, which is located at BE about 400 eV, points out to the presence of some nitrogen compound owing to co-deposition of the complexing agent in the deposit during electrolysis.

The spectral reflectance of black chromium deposited on the steel substrate is presented in Figs. 5 and 6. The influence of annealing temperature on the optical properties of samples was studied in the solar radiation region (200–2500 nm), which is usually used for quantitative comparison of the solar absorption of samples. It is known [12] that the spectral reflectance in the infrared (IR) region is generally dictated by the undercoating metal layer. The spectra of both total (RD) and diffuse (D) reflectances in the UV–visible–near infrared region are shown in Figs. 5 and 6. Some difference between RD and D spectra is observed only for the as-deposited coatings (curves 1^1 and 1^2), whereas after annealing of the samples both RD and D spectra are in line (curves 2^1 and 2^2). It is evident that losses related to the diffuse scattering component have little effect on the values of spectral reflectance for the samples under investigation. It is seen that the coating annealed at 400 °C as well as the as-deposited one shows a smooth horizontal feature of the spectral reflectance and displays a much lower reflectivity than that of the sample annealed at 600 °C. The feeble peak near 1900 nm results from water vapour in the air and water absorbed by barium sulphate, which was used as a diffusive reflectance standard. Taking into account that the Cr–Zn–P deposits are composed of dendrite-like agglomerations of the fine metallic particles in the matrix of dielectric materials, the low reflectivity can be attributed to the metallic structure which traps the light and induces multiple reflections leading to enhanced absorption due to an increased path of the light inside the material. We assume that if the optical properties of the sample before and after annealing at 400 °C are close, there is no any considerable difference in their microstructures. The reflectance spectra recorded for the samples annealed at 600 °C differ substantially from those for both as-deposited and annealed at 400 °C coatings. Figures 5 and 6 show that after annealing at 600 °C the reflectance increased in the infrared region and at a wavelength of 2000 nm became at least two- or three-fold higher than that of both as-deposited and annealed at 400 °C coatings. The reflectance measurements were used to derive absorption which is the fraction of incident energy absorbed by the material. According to the literature data [13]

$$A + T + R = 1, \tag{1}$$

where A is the absorption, T is the transmittance, R is the reflectance of the surface.

As T = 0 for the opaque surface, the absorption is determined by the equation:

$$A = 1 - R. \tag{2}$$

On the average, the magnitude of the total reflectance of as-deposited black Cr–Zn–P coatings was found to be about 5% and it remained such after annealing at 400 °C with some deviation to higher reflectance values for samples plated at 0.3 A cm^{-2}. It can be said that the value of solar absorption is about 95%. However, after annealing of the samples at 600 °C the solar absorption was found to be almost unchanged in the visible range and about 75–85% in the infrared one. Analysis of the results gives an insight into the causes of degradation of the Cr–Zn–P coating subjected to an elevated temperature (600 °C), which is very important over the lifetime

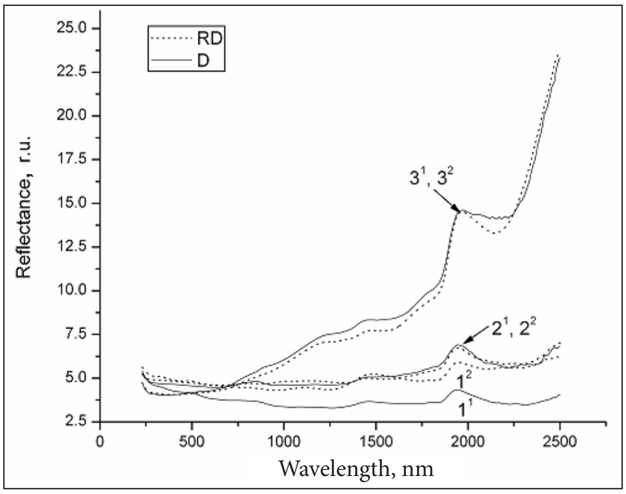

Fig. 5. The reflection spectra for the black Cr–Zn–P coating recorded before (1^1, 1^2) and after annealing of the sample at 400 °C (2^1, 2^2) and 600 °C (3^1, 3^2). The coating was deposited at 0.2 A cm^{-2}

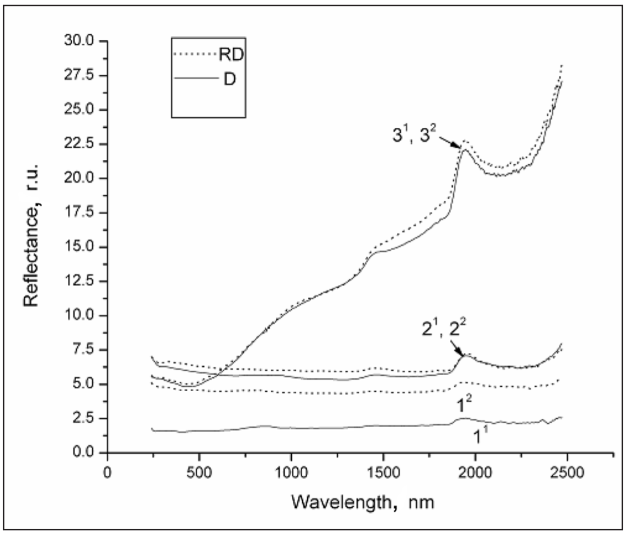

Fig. 6. The reflection spectra for the black Cr–Zn–P coating recorded before (1^1, 1^2) and after annealing of the sample at 400 °C (2^1, 2^2) and 600 °C (3^1, 3^2). The coating was deposited at 0.3 A cm^{-2}

of selective solar coatings. Thus, it is safe to say that the black Cr–Zn–P alloy deposited from the Cr(III) bath can be used as a good solar energy absorber.

CONCLUSIONS

Black Cr–Zn–P coatings were electrodeposited on the steel substrate from the trivalent chromium bath using glycine as a complexing agent, ZnO as a second main component and sodium dihydro-phosphate as a source of phosphorus.

The relationship between the microstructure and optical properties of the black Cr–Zn–P coating deposited on the steel substrate has been studied using as-deposited and annealed samples. The deposits were found to have nodular crystals of various sizes with microcracks undergoing changes during heat treatment. XRD patterns suggest that Cr–Zn–P alloy is a solid solution, which undergoes a series of transformations during annealing at 600 °C. The XPS results suggest that the near-surface layer is rich in Cr and Zn hydroxides, whereas oxides become dominant towards the substrate/coating interface. Besides, small amounts of phosphor and organic substances are also present in the coating. The phase constitution of black chromium varies by sample heating up to 600 °C. The black Cr–Zn–P coating has good optical properties and these properties remain practically constant after annealing of samples at a temperature of 400 °C. It has been found that sample annealing at 600 °C initiated an increase in reflectance caused by the changes in the structure and chemical composition of the deposit and the diffusion of the substrate material (Fe) into black chromium.

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ELEKTROCHEMIŠKAI NUSODINTŲ IR ATKAITINTŲ Cr–Zn–P DANGŲ, GAUTŲ IŠ TRIVALENČIO CHROMO ELEKTROLITO, APIBŪDINIMAS

Santrauka

Juodo chromo danga elektrochemiškai nusodinta ant plieno iš Cr(III) elektrolito, kurio sudėtyje yra ZnO ir NaH_2PO_4 $2H_2O$. Ištirta aukštos temperatūros įtaka dangos sudėčiai, struktūrai, morfologijai ir optinėms savybėms. Šiems tyrimams atlikti naudotos RSD, SEM ir REFS metodikos. Juodo chromo atspindžio koeficientas matuotas šviesos bangų ilgių 200–2 500 nm intervale. Pavyzdžiai su juodo chromo danga buvo kaitinami 2 val. oro atmosferoje 400 ir 600 °C temperatūrose. Nustatyta, kad prieš pavyzdžių atkaitinimą viršutiniuose dangos sluoksniuose vyrauja abiejų metalų hidroksidai su nedideliu kiekiu fosforo, tačiau po dangos atkaitinimo vyrauja abiejų metalų oksidai su fosforu ir nedideliu kiekiu metalinio Cr, kuris atsiranda kaitinimo metu (600 °C). Optiniai matavimai parodė, kad Cr–Zn–P danga pasižymi geromis optinėmis savybėmis, kurios beveik nesikeičia netgi esant 400 °C temperatūrai. Tačiau, pakėlus kaitinimo temperatūrą iki 600 °C, šios dangos absorbcinės savybės pablogėja dėl dangos cheminės sudėties, struktūros pokyčių ir substrato (Fe) difuzijos į dangą. Šių tyrimų rezultatai leidžia manyti, kad juoda Cr–Zn–P danga galėtų būti naudojama saulės kolektorių gamyboje.