

Utilization of alternating current methods for manufacture of selective absorbing coatings for heat collectors

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Electrochemical processes, taking place during alternating current treatment of aluminum, and dependence of morphology, chemical composition and electrical properties of obtained surface on treatment regimes have been studied. The conditions, providing the formation of selectively absorbing Al/Al₂O₃ coating in hydrochloric acid solutions, have been determined. To improve the optical characteristics of such coatings we have elaborated an electrochemical alternating current deposition of cupric oxide on their surface in form of isolated CuO nanoparticles. The method is environmentally safe and does not require any re-equipment. It provides an enhanced spectral selectivity due to increase of sunlight absorption in visible spectral region without a significant emissivity increase in the infrared one.

Изучены процессы, протекающие при электрохимической обработке алюминия переменным током. Исследована зависимость химического состава, морфологии и электрических свойств поверхности от режима обработки и определены условия, при которых в солянокислых растворах образуются селективные поглощающие покрытия Al/Al₂O₃. Для улучшения оптических характеристик таких покрытий разработан электрохимический переменноточковый метод нанесения на их поверхность оксида меди в виде отдельных наночастиц CuO. Метод безопасен с экологической точки зрения, не требует замены технологической оснастки и приводит к увеличению спектральной селективности покрытий за счет роста коэффициента поглощения в видимой области спектра без заметного возрастания излучательной способности в инфракрасной области.

When developing the solar heat collectors, the a great attention is given to the application of effective selectively absorbing coatings which could have absorptivity for visible sunlight A_s near 1 and emissivity in infrared region $\varepsilon \rightarrow 0$ [1, 2]. Such selective coatings, especially if they were intended for use in flat non-vacuumed collectors, are evaluated from the viewpoint of

adhesion to substrate material, cost, stability against sunlight, elevated temperatures and humidity, and ecological safety of manufacture processes. The most often used deposition methods for selectively absorbing coatings are direct-current electrodeposition of chrome and nickel blacks. As substrates, copper, aluminum or stainless steel are used.

As an alternative, black cupric oxide (CuO) selectively absorbing coatings have been proposed that are obtained by chemical deposition of CuO films [2] or by hydrolysis of copper salts sprayed onto hot substrates [3]. However, because the spectral selectivity A_s/ε for obtained CuO coatings is insufficient [2], it is recommended to use this oxide only as a single layer in multilayered selectively absorbing coating [3]. As another alternative, it has been proposed [3, 4] to form a selectively absorbing surface by electrochemical anodizing to obtain about 100 nm thick films of black oxides on the metal surface. Such anodized selectively absorbing coatings adhere well to the substrate. To increase A_s of these selective coatings, the pores in anodic films adjacent to substrate are filled with metal particles absorbing visible light, using the electrochemical introduction thereof. For such electrochemical pigmentation, the solutions of copper, nickel and tin salts were used. To reduce the emission in IR region, ε , an antireflective layer, for example, tin dioxide, is applied onto the coating surface. A more simple way to obtain selectively absorbing Al/Al₂O₃ coatings by alternating current electrochemical treatment of aluminum surface is known [5]. The process is realized in HCl solutions (2–5 %) at a high current density ($j = 0.37$ A/cm²). This method is not widely used. In this work, we have made attempts to improve of that method with the aim to meet the ecological requirements as such as to improve the optical characteristics of selectively absorbing coatings. We have studied the mechanisms of electrochemical processes running during the alternating current treatment of aluminum in conditions described in [5]. Then we have investigated dependence of chemical composition, surface morphology, and electrical properties of obtained Al/Al₂O₃ layers on the deposition regimes. To enhance the optical characteristics of selectively absorbing Al/Al₂O₃ coatings, we have elaborated an electrochemical alternating current method to deposit an additive CuO layer on the surface thereof. The method is environmentally safe and does not require any re-equipment.

For electrochemical treatment, we used 30–50 μm thick aluminum foil samples each having 10 cm² surface area; from here on, the samples are referred to as working electrodes. The working electrode back side was electrically isolated by glued dielectric film. As a current source, a generator of alter-

nating sinusoidal symmetric currents was used, the current frequency ν being variable in the range of 50–150 Hz and the amplitude I in interval ± 10 A. The electrochemical cell consisted of a 700 ml thermostated glass vessel containing a working electrode, a stainless steel counter-electrode of 600 cm² area and saturated Ag/AgCl reference electrode of standard design, the working electrode potential U being determined relatively to the reference one. The electrolyte temperature was constant (20°C).

As electrolytes for alternating current treatment of aluminum to obtain selectively absorbing Al/Al₂O₃ coatings 0.5–5 % hydrochloric acid solutions were used. Besides, aluminum was treated in 1 % H₂SO₄. The current density j was varied from 0.005 to 1.0 A/cm². For subsequent alternating current electrochemical introduction of copper ions into Al/Al₂O₃ structure, aluminium oxide film blacking was used similar to [6]. To that end, we used the above described current source and electrochemical cell. As the electrolyte, sufficiently a solution containing 1 % H₂SO₄ and 0.2 % CuSO₄ was used, i.e. at concentrations much lower than those in [6]. The blacking was realized at frequency $\nu = 120$ Hz and working potential $U = 175$ V during 3 min.

To study the mechanism of the electrochemical processes, chronopotentiometry with controlled alternating current [7] was used that provided the recording the time dependence of working potential in the alternating current circuit-oscillographic chronopotentiogram (U - τ -curve) on the oscillograph screen. Since the working electrode functioned as cathode during one half-period of alternating voltage and as anode during another half-period, the reduction and oxidation stages were observed in chronopotentiometry with controlled alternating current. According to [7], for reversible electrochemical processes, the time delays in anodic (right) and cathodic (left) branches of oscillographic chronopotentiograms had to take place at the same potential. On the contrary, for irreversible processes, either the anodic delay should move to less negative potentials and cathodic delay to more negative ones, or one of the delays should be absent. So, the full U - τ -curve contained information on the nature of electrochemically active compounds (delay potential), their concentrations (delay duration), and reversibility of the electro-

chemical process (mutual arrangement of the cathodic and anodic delay potentials).

The optical properties of the obtained objects were analyzed by a reflection photometer FM-59 that allowed to determine A_s in the wavelength range $0.3 \mu\text{m} \leq \lambda \leq 2.4 \mu\text{m}$, and by thermoradiometer TRM "I" measuring ε in the wavelength range $4 \mu\text{m} \leq \lambda \leq 40 \mu\text{m}$. The breakdown voltages of the coatings U_b were studied by a EMG-1579-102 TR Orion characteriograph with the molybdenum probe polished hemispherical surface of $100 \mu\text{m}$ radius. Morphology of the samples was studied using a JSM-820 electron microscope [8]. The composition of about 10 nm thick sample sublayer was determined by X-ray photoelectron spectroscopy (XPS) on a XPS-800 Kratos spectrometer using ratio of photoelectron spectra areas of C1s, O2s and Cu3p, Al2p core levels taking into account their sensitivity factors [8, 9]. The binding energy was calibrated using C1s ($E_b = 285 \text{ eV}$) line as a reference. To determine the copper valence, the shape of Cu 2p core-level spectra was analyzed.

The mechanisms of electrochemical processes running in hydrochloric and sulfate electrolytes have been studied comparatively. If the electrolyte did not contain chloride ions, for example, if aluminium was treated by sinusoidal alternating symmetric current at a frequency ν of 50–150 Hz in 1 % H_2SO_4 , the oscillographic chronopotentiograms, in accordance with [7], demonstrated irreversible character of electrochemical processes (Fig. 1a). Neglecting the differences in absolute U values, which increased monotonously in 1 % H_2SO_4 during electrochemical treatment, the cathodic (left) branches of oscillographic chronopotentiograms in sulfate electrolyte are similar to those recorded in hydrochloric electrolyte (Fig. 1a–b), because they had a single delay. As far as value of such delay grew in accordance with increasing of acid concentration from 0.5 % to 5 %, we have concluded that it corresponds to the standard electrochemical reduction of hydrogen. The anodic process in sulfate electrolyte qualitatively differs from that in hydrochloric acid. As it is seen in Fig. 1a, the anodic (right) branches of oscillographic chronopotentiograms in sulfuric acid solution show only one delay that could be corresponded to the process

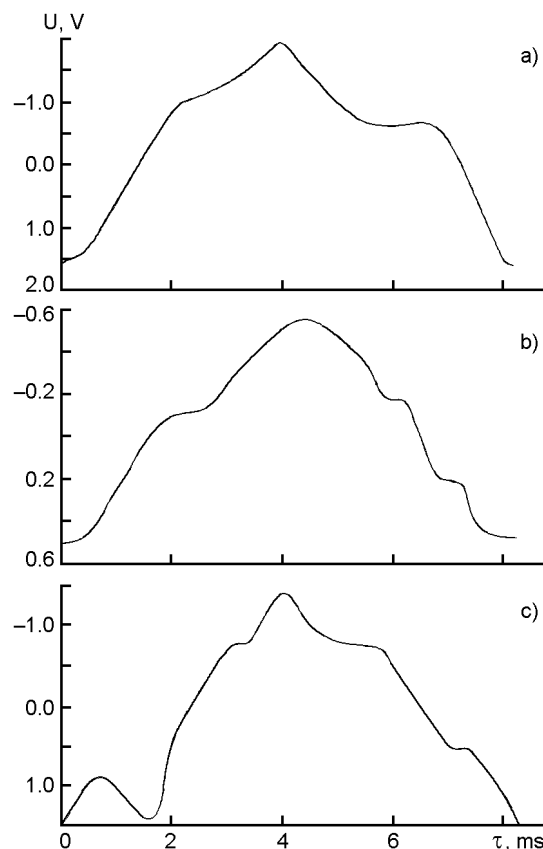
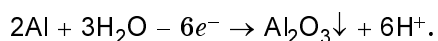


Fig. 1. Oscillographic chronopotentiograms (U - τ -curves) of alternating current electrochemical treatments of Al (a,b) and electrochemical pigmentation of $\text{Al}/\text{Al}_2\text{O}_3$ (c) in regimes: 1 % H_2SO_4 , $\nu = 120 \text{ Hz}$, $j = 0.02 \text{ A/cm}^2$ (a); 0.5 % HCl , $\nu = 120 \text{ Hz}$, $j = 0.1 \text{ A/cm}^2$ (b); 1 % H_2SO_4 + 0.2 % CuSO_4 , $\nu = 120 \text{ Hz}$, $j = 0.05 \text{ A/cm}^2$ (c).

In 1 % H_2SO_4 electrolyte, no etching of the aluminum surface is observed, on the contrary, the growing of oxide film takes place over the whole surface. The continuity and high electric resistance of the films are confirmed by breakdown voltage values $U_b \geq 50 \text{ V}$ for $\text{Al}/\text{Al}_2\text{O}_3$ films obtained by alternating current treatment at $U = 175 \text{ V}$ in 1 % H_2SO_4 during 10 minutes.

The oscillographic chronopotentiogram shown in Fig. 1b is typical of processes running in 0.5–5 % HCl within the whole studied frequency range. It contains a single delay in the cathodic branch and two delays in the anodic one, moreover, the potentials of cathodic and anodic delays have different values, thus evidencing that these processes are irreversible. The observed U - τ -curve shape is explained by the fact that, in accordance with [10], as an alternating current flows through hydrochloric acid solu-

tion, some products of chloride-ions discharge are formed in anodic half-period. In our opinion, the formed hypochlorite and chlorate-ions cause aluminum oxidation. The aluminum surface treated by alternating current in hydrochloric acidic solution is etched on some areas due to formation of poorly soluble AlCl_3 salt, but on the others, it becomes coated by insoluble oxide Al_2O_3 . This supposition is confirmed by X-ray photoelectron spectroscopy examinations, which have shown that such surface had commensurable amounts of aluminum and oxygen ions but only traces of chlorine.

The morphology examinations of the surfaces obtained by different alternating current treatments at $\nu = 50\text{--}150$ Hz in 0.5–5 % HCl have shown that at all frequencies, the increasing acid concentration, as well as electrolysis duration and current density, results in growing surface micro-relief. The latter, according to optical measurements, agrees with increasing A_s values. Fig. 2a shows the black $\text{Al}/\text{Al}_2\text{O}_3$ coating prepared by alternating current treatment in 5 % HCl at $\nu = 75$ Hz and $j = 1.0$ A/cm^2 during 2 min. The breakdown voltage U_b for different areas of this coating varies from 0.5 V to 9.0 V. The coating has high-developed surface relief, the micro-roughness exceeding 10 μm . The absorptivity of such coating is sufficiently high ($A_s = 96$ %). At the same time, it exhibits a high emissivity in IR region, namely $\varepsilon = 0.74$, what is associated, in our opinion, with the relief which is commensurate with infrared light wavelengths. Due to the latter fact, the coating is almost spectrally unselective.

Although j decreasing results in a worse visible light absorption, it causes a considerably reduced emission in IR region that corresponds, to some extent, to increasing A_s/ε . The dilution of hydrochloric acid also causes a reduced ε along with the reduction of A_s . A similar influence is due to decreasing of electrochemical treatment duration. For example, if HCl concentration is 1 % and current density j is 0.1 A/cm^2 , it is possible to obtain at frequency $\nu = 120$ Hz during 3 min (regime 1) black coatings having $A_s = 0.90$ and $\varepsilon = 0.58$, and during 1 minute (regime 2), grey selective coatings with optical parameters $A_s = 0.70$ and $\varepsilon = 0.16$, for which spectral selectivity A_s/ε equals 4.4. Electron-microscopic photo of the surface prepared in regime 2 (shown on Fig. 2b), demonstrates the surface relief less developed as compared to that of Fig. 2a. Ac-

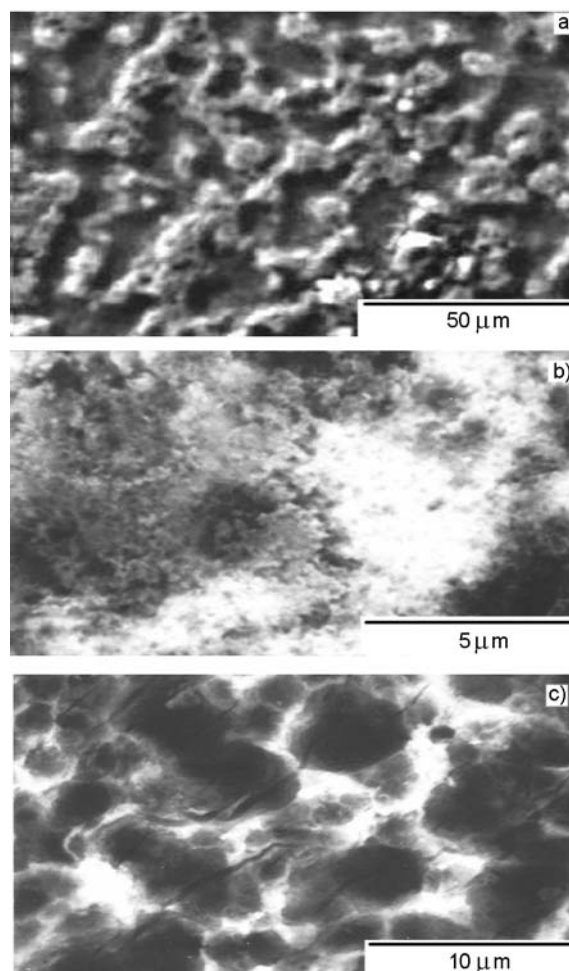


Fig. 2. Electron-microscopic photos of Al surfaces, electrochemically treated by alternating current in regimes: 5 % HCl, $\nu = 75$ Hz, $j = 1.0$ A/cm^2 ; $\tau = 2$ min; optical parameters: $\alpha_c = 0.96$; $\varepsilon = 0.74$ (a); 1 % HCl, $\nu = 120$ Hz, $j = 0.1$ A/cm^2 ; $\tau = 1$ min; optical parameters: $\alpha_c = 0.70$; $\varepsilon = 0.16$ (b); 0.5 % HCl, $\nu = 120$ Hz, $j = 0.1$ A/cm^2 ; $\tau = 1$ min; optical parameters: $\alpha_c = 0.42$; $\varepsilon = 0.10$ (c).

ording to Fig. 2b, the film prepared in regime 2 is characterized by micro-roughness up to 5 μm height which contain fine particles of several hundreds nanometers size, i.e. commensurable with visible light wavelengths. For such films, the breakdown voltage is to 10 V, evidencing existence of thin dielectric oxide film on the aluminum surface.

An further considerable decrease of hydrochloric acid concentration at the same current density during the electrochemical treatment influences negatively the coating selectivity. For example, if HCl concentration was 0.5 % and frequency was 120 Hz, the electrochemical treatment during 1 min

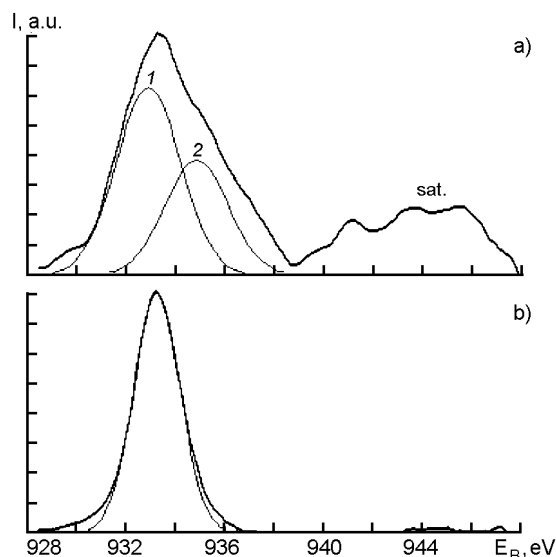


Fig. 3. $\text{Cu}2p_{3/2}$ XPS-spectra of the Al sample electrochemically treated by alternating current during 10 min in 1 % H_2SO_4 at $\nu = 120$ Hz and $U = 175$ V, and then electrochemically pigmented. The *3a* spectrum was obtained during first 100 s of the X-ray exposure, the *3b* spectrum, after 40 min of the exposure.

results in a coating poorly absorbing in the visible part of the spectrum with $A_s = 0.42$ and $\varepsilon = 0.10$. The film has a well developed surface relief with rounded cavities about $5 \mu\text{m}$ in diameter. It is typical that fine particles of sub-micrometric dimensions are absent, that, in our opinion, is a cause of its small absorption of short-wavelength (visible) light. Electrical measurements gave values of breakdown voltage $U_b > 100$ V, meaning the presence of a thick Al_2O_3 layer on the surface of such coating. By decreasing of current density values to $j = 0.03$ – 0.06 A/cm^2 in the case of electrochemical treatment of Al in 0.5 % HCl at the same frequency $\nu = 120$ Hz during 10 min (regime 3) we have obtained thinner Al_2O_3 films and gray coatings with $A_s = 0.50$, $\varepsilon = 0.11$.

In general, varying of parameters at the alternating current electrochemical treatment of aluminium in hydrochloric acid solutions did not allowed us to obtain optical selective coating with spectral selectivity $A_s/\varepsilon > 4.5$, what is in agreement with literature data [3, 5]. So we have decided to enhance the absorption of sunlight by gray Al/ Al_2O_3 layers by alternating current electrodeposition of CuO films on Al/ Al_2O_3 surface in diluted copper sulfate solution. It is to note that alternating current electrochemical pigmentation of aluminum oxide

films, associated by authors [5, 6] with introduction of copper particles into Al_2O_3 pores, was utilized in much more concentrated acidic solutions. According to [10], sinusoidal alternating symmetric current treatment results in deposition of compact Cu coatings on conducting substrates from electrolyte containing CuSO_4 and H_2SO_4 .

However, in very diluted solutions of CuSO_4 (0.2 %) containing only 1 % H_2SO_4 , the alkalizing of near-electrode space along with accumulation of cuprous ions (Cu^+) in the electrolyte volume may result in deposition of copper oxides and hydroxides on the surface of working electrode [6]. To distinguish the mechanism of electrochemical processes during the pigmentation in very diluted acidic copper sulfate solutions, and to analyze the physical-chemical nature of pigmentation products, we investigated the films treated previously by alternating current in 0.5–5 % HCl or in 1 % H_2SO_4 . Oscillographic chronopotentiograms of the alternating current electrochemical pigmentation process (Fig. 1c) revealed their irreversibility. The shape of U - τ -curves is independent of the nature of solutions in which the previous alternating current treatment of Al was realized (i.e. in 0.5–5 % HCl or in 1 % H_2SO_4), that allows to suppose the identity of the pigmentation processes and products for both types of Al/ Al_2O_3 surfaces.

The layer-by-layer analysis of the layers obtained by alternating current pigmentation of surfaces treated previously in solutions of hydrochloric and sulfuric acids, also revealed their physical-chemical identity. Fig. 3 presents $\text{Cu}2p_{3/2}$ photoelectron spectra of the sample electrochemically treated by alternating current during 10 min in 1 % H_2SO_4 at frequency $\nu = 120$ Hz and $U = 175$ V, and then electrochemically pigmented. The *3a* spectrum is taken after first 100 s, the *3b* spectrum, after 40 min of the X-ray exposure. The spectrum in Fig. 3a has a main line and a satellite structure ($E_e \approx 943$ eV), typical of Cu^{2+} compounds [11]. This is well-known "shake-up" satellite attributed to charge transfer from ligand to unoccupied $3d$ Cu level in the final state of photoemission. It is absent in spectra of Cu^+ compounds. The intensity of this peak is about 50 % of the main line in CuO spectrum. The main line in Fig. 3a can be subdivided into two lines that differ in binding energy by about 2 eV. Line 1 has binding energy typical of Cu_2O but line 2 can be

ascribed both to CuO and to Cu(OH)₂. It is clear from Fig. 3 that Cu²⁺ under X-ray irradiation during the exposure in ultrahigh vacuum transforms into Cu⁺, because the spectrum in Fig. 3b is typical for Cu₂O. We have not found in literature any data on decomposition of massive cupric oxide samples under X-ray irradiation. Such behavior of CuO film can be explained by peculiarity of its structure, which allows easy reduction as a result of X-ray irradiation [12]. Quantitative analysis of surface composition shows (see Table) that cupric oxide film is non-continuous, since aluminum lines (in Al₂O₃ state) have a rather high intensity. Moreover, signals from sulfur are absent in the spectrum. The Table also demonstrates the surface composition of this sample after removing of 50 nm thick surface layer by ion bombardment (Ar⁺, *E* = 2 keV). It is clear that sputtering of such surface layer practically removes hydrocarbon contamination and adsorbed oxygen, but did not decrease relative concentration of copper. Independent of chemical nature of the electrolytes, the particles obtained by electrochemical pigmentation were consisted mainly of CuO and were deposited as a rule in pores of Al₂O₃ films. The Al₂O₃ surface remained partly uncovered with copper oxides. Electron-microscopic photo (Fig. 4a) of the sample having the XPS-spectrum shown in Fig. 3a and atomic composition presented in the Table demonstrates smooth (within 20 nm of resolution) surface with isolated white, i.e. having large emissivity, particles of CuO — more conductive material than Al₂O₃ [13] filling the pores of the anodic film.

Electrochemical deposition of CuO allowed to enhance the absorption of sunlight (short-wavelength radiation) in the coating without significant increasing of its emission in IR region. Fig. 4b shows electron-microscopic photo of surface treated in 0.5 % HCl at *j* = 0.03–0.06 A/cm² and *v* = 120 Hz during 10 min (regime 3) fol-

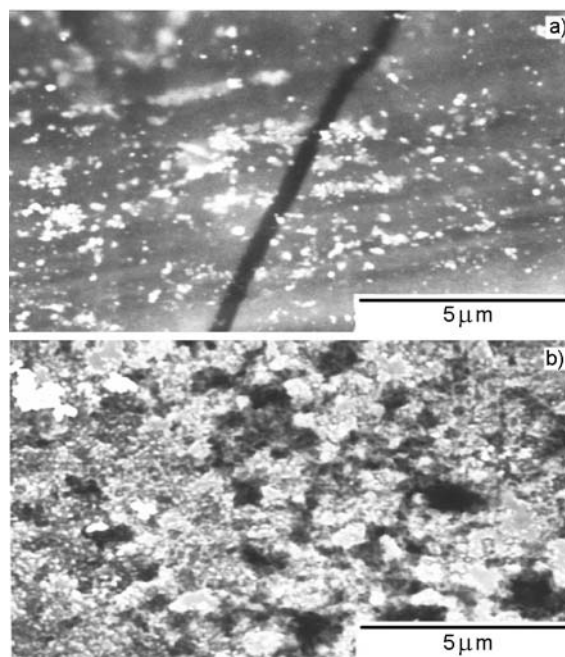


Fig. 4. Electron-microscopic photos of Al/Al₂O₃ surfaces electrochemically pigmented in solution 1 % H₂SO₄ + 0.2 % CuSO₄. The surfaces were obtained by alternating current electrochemical treatment of Al in regimes: 1 % H₂SO₄, *v* = 120 Hz, *j* decreases monotonously at *U* = 175 V, *τ* = 10 min (a); 0.5 % HCl, *v* = 120 Hz, *j* = 0.03–0.06 A/cm²; *τ* = 10 min (b). Optical parameters of Al/Al₂O₃ surface prior to pigmentation: $\alpha_c = 0.50$; $\varepsilon = 0.11$; optical parameters after CuO deposition on Al/Al₂O₃: $\alpha_c = 0.82$; $\varepsilon = 0.115$.

lowed by electrochemical pigmentation in diluted acidic solution of copper sulfate. As it is seen in Fig. 4b, the result of alternating current electrochemical pigmentation is multiphase and different scale fine-cellular surface. The similar size and contrast of white particles in Fig. 4a and Fig. 4b give evidence to their identical nature, thus, isolated islands of CuO electrodeposited on the aluminum surface preliminarily etched in

Table. Atomic concentrations of elements on surface of Al beforehand electrochemically treated by alternating current during 10 min in 1 % H₂SO₄ at frequency *v* = 120 Hz and then electrochemically pigmented in solution consisted from 1 % H₂SO₄ + 0.2 % CuSO₄ at frequency *v* = 120 Hz and potential of working electrode *U* = 175 V during 3 min

Sample Al/Al ₂ O ₃ /CuO	Elemental composition, at. %			
	C	O	Al	Cu
Surface as-prepared	20.8	50.4	25.9	2.9
Surface after removing of 50 nm thick layer by ion bombardment	4.0	45.2	46.0	4.8

diluted HCl solution (Fig. 4b) gives rise to micro-roughness of several hundreds of nanometers size, i.e. of same order as visible light wavelengths. The light absorption inherent in CuO and formation of additive micro-roughness of cupric oxide particles commensurate with visible light wavelengths alongside with micro-roughness of Al/Al₂O₃, in our opinion, is resulted in increased absorption of sunlight ($A_s = 0.82$), that, in combination with low emissivity ($\varepsilon = 0.115$) for film presented in Fig. 4b, provides a high spectral selectivity $A_s/\varepsilon = 7.1$.

Thus, in this work, determined have been the mechanisms of alternating current electrochemical processes due to which aluminum surfaces are partly etched and partly coated by oxide Al₂O₃ during treatment in diluted solutions of hydrochloric acid, and therefore selectively absorbing Al/Al₂O₃ coatings are formed. The electrochemical pigmentation of Al/Al₂O₃ coatings by alternating current treatment in diluted acidic solution of copper sulfate is associated with electrodeposition of isolated CuO islands on the electrochemically treated aluminum surface, due to which additive nanoparticles of several hundreds nanometers size are formed. This provides an enhanced absorption of sunlight without significant increase of the emissivity in infrared region.

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Використання зміннострумових електрохімічних методів з метою виготовлення селективних поглинаючих покриттів теплових колекторів

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Вивчено процеси, які відбуваються протягом електрохімічної обробки алюмінію змінним струмом. Досліджено залежність хімічного складу, морфології та електричних властивостей поверхонь від режимів обробки і визначено умови, за яких в солянокислих розчинах можуть бути виготовлені селективні поглинаючі покриття Al/Al₂O₃. З метою поліпшення оптичних властивостей таких покриттів розроблено зміннострумовий електрохімічний метод нанесення на їх поверхню оксиду міді у формі окремих наночастинок CuO. Метод є безпечним з екологічного погляду, не потребує заміни технологічної оснастки і приводить до збільшення спектральної селективності покриттів внаслідок підвищення коефіцієнта поглинання у видимій частині сонячного спектра без помітного зростання здатності до випромінювання в інфрачервоній частині.