



Research article

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## Functional indium oxide-based thin-film coatings with variable light transmission

D.A. Strekalovskaya , L.P. Baturova , A.V. Semencha , E.K. Aleksandrova 

Peter the Great St. Petersburg Polytechnic University, St. Petersburg, Russia

✉ [darya.strek@gmail.com](mailto:darya.strek@gmail.com)

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**Abstract.** One of the directions of energy-saving technologies development in the public works construction is associated with the engineering of glazing materials that allow regulating light and heat flux passing through them. Known solutions are associated with the use of multilayer materials with special optical thin-film electrochromic coatings. This work presents the results of the functional properties study of single-layer transparent conductive thin-film coatings based on tin-doped indium oxide (ITO films), formed on a glass substrate by magnetron sputtering. It is shown that, under conditions of cathodic treatment in a potentiodynamic mode at certain threshold potentials  $E_p$  in aqueous and aqueous-ethanol solutions, the introduction of hydrogen atoms into surface layers is observed, leading to a decrease in the transmittance  $T_x$  to ~10% and the transition of ITO films to a colored state. It was found that the introduction of hydrogen atoms is accompanied by the formation of hydrogen-indium bronzes with the general formula  $H_xIn_2O_3$ , the composition of which depends on the pH of the solution. Indium bronzes formed in a neutral 3% NaCl solution are characterized by a low value of the x index, high conductivity and reversibility of the coloration-discoloration processes, i.e. have electrochromic properties. Indium bronzes formed in 1M aqueous and aqueous-ethanol KOH solutions have higher values of the x index, low conductivity and irreversibility of the coloring process. Increase bronzes conductivity due to the introduction of carbon nanoparticles into the outer layers of ITO films does not affect the reversibility of the coloration-discoloration processes. The results obtained can serve as a basis for the creation of a wide range of modern improved glass and double-glazed windows with an adjustable value of light transmittance.

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### 1. Introduction

One of the urgent tasks in modern energy-efficient buildings construction is to create conditions for protection from environmental influences [1]–[3]. It is often necessary to reduce the excess thermal solar radiation (TSI) penetrating into living spaces through glazing elements [4]–[6]. To reduce the TSI value and to have the ability to control the degree of illumination in a room, glazing materials must have a set of properties capable of directionally changing their parameters: transmitting a light flux in a discoloration state or inhibiting the transmission intensity in a coloration state. Usually multilayer transparent thin-film coatings

with special properties are used to impart desired functional properties to the glazing materials [7], [8]. An integral part of such coatings is the presence of an electrochromic layer formed on a conductive transparent substrate (current-conductive layer), capable of reversibly changing optical properties (transmission) during alternating polarization. Thus, the use of glazing materials with multilayer electrochromic coatings, which reversibly change the light transmission coefficient, makes it possible to reduce energy costs for cooling, heating or room lighting [9]–[11].

Thin-film coatings based on transition metal oxides (TMO) exhibit electrochromic properties and, due to their high optical transparency in the visible wavelength range and electrical conductivity, are widely used in various fields of electronic and microsystem engineering [12]–[15]. The thin films functional properties are determined primarily by their composition and structure [16]–[18]. Upon contact of thin-film coatings based on transition metal oxides with an electrolyte under conditions of cathodic polarization, adsorbed hydrogen atoms  $H_{ads}$  formed during the discharge of water molecules:  $H_2O + e \rightarrow H_{ads} + OH^-$ , can accumulate on the surface and be intercalated into the oxide. The introduction of adsorbed hydrogen atoms into a transparent oxide can lead to the formation of compounds of various nonstoichiometric compositions with a general formula  $H_xM_yO_z$  and cause a change in its electrical and optical properties [19]–[21]. If the process of intercalation proceeds reversibly in the film, then upon changing the direction of polarization the film can restore its original properties. As materials with reversible properties, of particular interest are coatings based on indium-tin oxide (Indium Tin Oxide - ITO), which are widely used as a transparent conductive sublayer in the creation of various indicator systems, monitoring sensors, electrochromic devices with variable optical density, etc. [22]–[25].

The object of the study was transparent electrically conductive thin films of tin-doped indium oxide in a ratio of 90% indium oxide  $In_2O_3$  and 10% tin oxide  $SnO_2$  (ITO-films) laid on a glass substrate by magnetron sputtering. The research purpose was to study the optical properties of thin ITO films in aqueous and aqueous-ethanol solutions with different pH values in a wide range of potentials and to determine the factors leading to a change in its functional properties. In this work, we used solutions characterized by different values of hydrogen evolution overvoltage in which the dissolution of ITO films is minimized. For this purpose, analysis of Pourbaix diagrams for In- $H_2O$  and Sn- $SnO_2$  systems was preliminary performed and pH interval was found in which  $In_2O_3$  and  $SnO_2$  oxides are thermodynamically stable in aqueous media [19], [24]. The objectives of the study were the deposition of indium tin oxides-based thin films, electrochemical measurements by linear anodic and cyclic polarization, modification of indium oxide-based coatings, and evaluation of thin film coating optical properties – transmission coefficient. The obtained results make it possible to determine the prospects of using ITO films for glazing materials.

## 2. Methods

Thin-film coatings based on indium-tin oxides with a ratio of  $In_2O_3$ -90% and  $SnO_2$ -10% oxides were settled-down on transparent glass substrates by magnetron sputtering using the industrial vacuum spraying plant "Aspira-150" (Research and manufacturing complex "Polytech"). The transmission coefficient  $T_0$  of the initial ITO films with a thickness of 0.21  $\mu m$  with a uniformity of  $\pm 0.4\%$  was 83%.

The current-voltage characteristics of ITO films were compared in aqueous and aqueous-ethanol solutions with different pH values. Cyclic voltage curves (CVC) were taken in a potentiodynamic mode using the IPC-PRO M potentiostat of the Volta company with a computer system for recording and processing data. The potential sweep rate  $V_p$  was 1  $mV \cdot s^{-1}$ . The reference electrode was a saturated silver chloride electrode (all values of potentials  $E$  are given relatively to standard hydrogen electrode (SHE)). Ethyl alcohol had the reagent grade qualification.

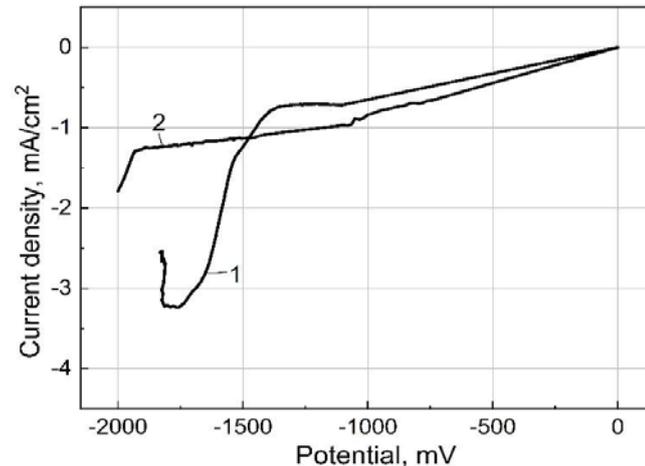
The installation allowed to take absorption spectra of ITO films at different potentials simultaneously with electrochemical measurements, without removing the sample from the solution. For this purpose, a compact USB-650 Red Tide spectrometer was used. It was fixed in such a way that the illuminator's light beam passed through the cell, the electrolyte solution and the working thin-film electrode. The reference electrode was located outside the cell in a separate vessel with a saturated potassium chloride solution and connected to the system using a micro electrochemical key. Transmission coefficients  $T(\lambda)$  were determined in the wavelength range from 150 to 1000 nm at a wavelength  $\lambda = 550$  nm.

During studying the elemental composition of ITO films surface after cathodic treatment in a potentiostatic mode in a 1M KOH solution, containing hydroxylated water-soluble derivatives of fullerenes – fullereneol  $C_{60}(OH)_{24}$ , two-layer films were used, obtained by alternating the ITO layer and the nickel oxide (II) layer. The NiO layer was applied by chemical deposition from the gas phase in an argon-oxygen medium with small ozone additives ( $Ar + 5 \text{ vol.}\% O_2 + 0.1 \text{ vol.}\% O_3$ ) at temperature of 330°C, using a horizontal quartz reactor with "hot walls" equipped with a gas inlet system, a furnace with a thermostat, a vacuum meter in a gas medium [16]. The elemental composition of the ITO films outer layers was studied

by X-ray photoelectron spectroscopy (XPS) using the Nano-FAB 25 installation jointly produced by NT-MDT (Russia) and SPECS (Germany). The analyzer sensitivity is 0.1%, the penetration depth during the analysis reached 10 nm, the analysis area is 0.1 mm<sup>2</sup>. The analysis of X-ray photoelectronic spectra and their decomposition was carried out using special Casa XPS software (version 2.3.18, Casa Software Ltd, 2020 [26]).

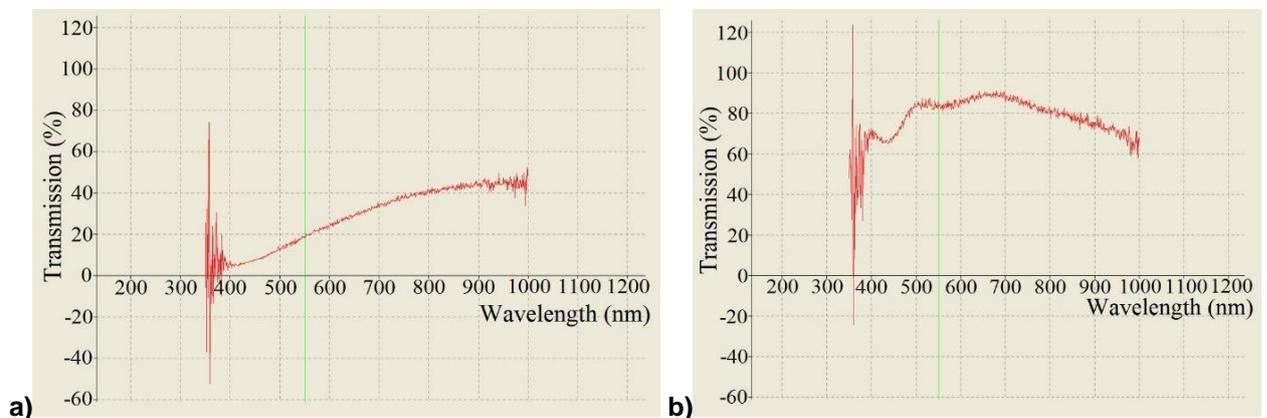
### 3. Results and Discussion

Thin-film devices typically use ion-conducting electrolytes with a wide range of electrochemical stability. The ITO films behavior under polarization conditions was studied in aqueous and aqueous-ethanol 1M KOH solution, as well as in 3% NaCl solution, in which indium oxide is thermodynamically stable. Fig. 1, Curve 1 shows the cathode branch of the ITO-film voltage curve obtained in an aqueous 1M KOH solution in the potential range from initial  $E_{in}$  to -1.85 V.



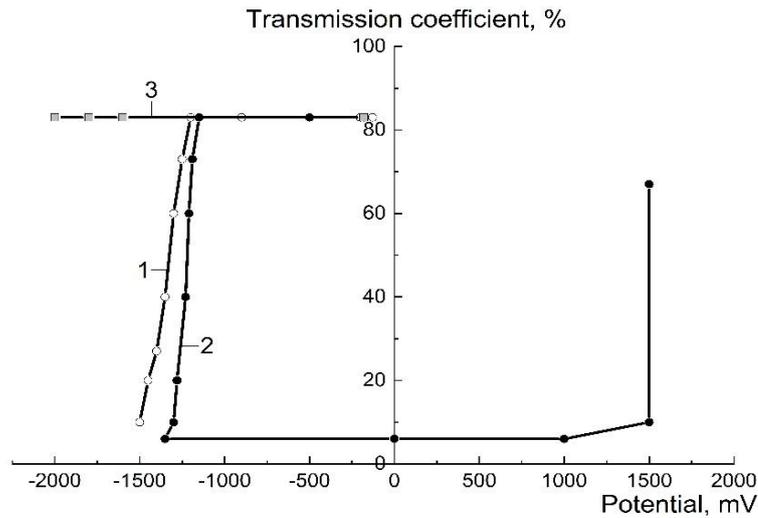
**Figure 1. Cathode branches of voltage curves obtained on an ITO film in an aqueous (1) and aqueous-ethanol (2) 1M KOH solution.**

At a potential of -1.45 V, an increase in the cathode current is observed on the polarization curve, due to the hydrogen release process beginning due to the discharge of water molecules and a change in the ITO film optical absorption. ITO films turn into a dark brown colored state. At a potential of -1.8 V, the current reaches its maximum value. A further potential shift in the negative direction leads to a decrease in the rate of the hydrogen release process due to an increase in the resistance of the film due to the process solid-phase diffusion difficulties. In accordance with this, it was not possible technically to obtain the ITO films anode branch in the painted state. The obtained results have demonstrated that after switching off polarization in an open circuit, ITO films retain their properties in the colored state. The transmission coefficient does not change for a long time (up to a day or more), i.e. ITO films have an "optical memory effect" and are characterized by non-volatility. The ITO films storage time in the air also does not affect the transmittance value. This made it possible to determine the ITO films transmittance coefficients in the colored state at different potentials under open circuit conditions. Figure 2, a shows the transmission coefficient spectrum obtained on an ITO film in the staining state at a potential of -1.85 V.



**Figure 2. Transmission spectra of ITO films obtained after record of volt-ampere curves (VAC) in aqueous (a) and ethanol (b) 1M KOH solution.**

The transmission  $T_x$  coefficient, determined at the wavelength  $\lambda = 550$  nm, was 18.4%. Figure 3, Curve 1 shows the dependence of the transmission coefficient  $T_x$  (%) of ITO films on the value of the potential  $E$ , obtained in 1M KOH solution in a wide range of potentials.



**Figure 3. Dependences of the ITO films transmittance on the potential obtained in various solutions: 1 – 1M KOH solution; 2 – 3% NaCl solution; 3 – 1M KOH solution in ethanol.**

As can be seen, the ITO films staining beginning potential (the potential of the transmission reduction coefficient beginning) is -1.45 V, which coincides with the potential of the VAC cathode current branch growth beginning. The transition from the discolored state to the state of complete ITO films staining in 1M KOH solution occurs in a fairly narrow range of potentials of the 50 mV order.

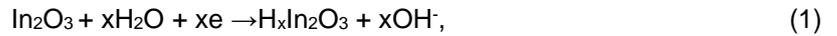
The cathode branch record of the voltampere curve on an ITO film in an anhydrous 1M KOH solution in ethanol (conditionally anhydrous solution containing no more than 0.5 wt% H<sub>2</sub>O) shows that there is no peak cathode current on the polarization curve in the studied potential range and a small limiting current associated with the reduction of dissolved oxygen is observed. Figure 2, b shows the transmission coefficient spectrum obtained on an ITO film with a final sweep potential of -2.0 V. The ITO film transmission coefficient, determined at a wavelength of  $\lambda = 550$  nm, was 83% (Figure 3, curve 3), which corresponds to the transmission coefficient of the original ITO films. This indicates that in the 1M KOH solution in ethanol, ITO films in a wide range of potentials are not electrochemically active by themselves and the transition from aqueous alkaline media to ethanol leads to their electrochemical stability window expansion.

When 5 wt% H<sub>2</sub>O is added into a 1M KOH solution in ethanol, a section associated with the hydrogen evolution process appears on the cathode branch of the VAC at potentials  $E \leq -1.8$  (Fig. 1, curve 2) and the ITO film staining in a dark brown color is observed (Fig. 4). The potential for staining beginning  $E_{stb}$  in an ethanol 1M KOH solution containing 5 wt% H<sub>2</sub>O is -1.8 V and shifts in comparison with an aqueous 1M KOH solution to the cathode side by 350 mV. This is due to the fact that the transition from aqueous alkaline media to ethanol media with a low water content is accompanied by an increase in the overvoltage of hydrogen release and a change in the nature of the delayed stage. So, if in an aqueous 1M KOH solution, the speed of the process is determined by the delayed stage of discharge of water molecules, then in ethanol with small water additives – by the recombination stage of adsorbed hydrogen atoms [27]–[29]. This leads to an increase in the degree of surface filling with adsorbed hydrogen H<sub>ads</sub> contributing to the transition of H<sub>ads</sub>→H<sub>ab</sub> and the addition of hydrogen into the ITO film surface layer. The  $T_x$  transmission coefficient, determined at a potential of -1.8 V in an ethanol solution containing 5 wt% H<sub>2</sub>O, decreases by about 2.5 times compared to an aqueous 1M KOH solution and reaches 7.6%.



**Figure 4. ITO film deposited on a transparent glass substrate in a colored state.**

Thus, under conditions of ITO films cathodic polarization in 1M KOH solution, a change in their optical and electrical properties is observed in the region of hydrogen release potentials: an intense dark brown film color appears and electrical conductivity decreases. A strong modification of ITO films properties in the region of hydrogen release potentials is due to the fact that the resulting atomic hydrogen, penetrating into ITO films surface layer, participates in the heterogeneous process of indium oxide reduction  $\text{In}_2\text{O}_3$  with the formation of hydrogen indium bronze with the general formula  $\text{H}_x\text{In}_2\text{O}_3$ :

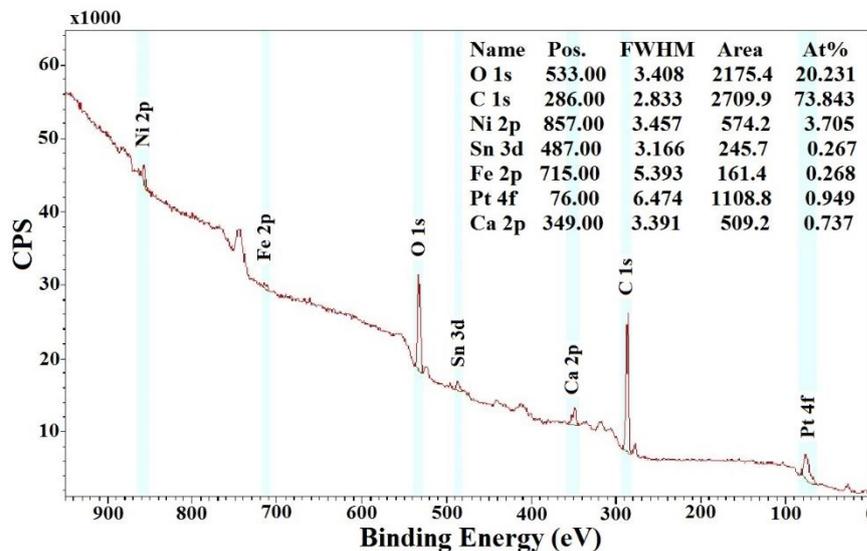


where  $x$ - is the stoichiometric coefficient varying in the interval  $0 < x < 1$ . During the hydrogen ion discharging process, the thickness of the indium bronze layer increases, a film with a "sandwich structure" is formed and ITO film sequential transition from a state with high electrical conductivity to a state with low one is observed. When the structural transformations, occurring during the bronze synthesis in the film end, the film goes into a stable state characterized by high insulating properties.

The reversibility of the staining-discoloration process of ITO films is determined by the hydrogen deintercalation degree from the indium bronze  $\text{H}_x\text{In}_2\text{O}_3$  layer in the process of recording the voltampere curve (VAC) anode branch. An increase in the electrochemical activity of indium bronze layers formed under conditions of the VAC cathode branch recording can be carried out by modifying their properties by introducing impurities of various elements into them.

The work investigates the carbon injection effect on the ITO films kinetic characteristics. The carbon was added into the thin-film systems outer layers by cathodic deposition in a solution containing a water-soluble form of fullerene derivatives ( $\text{C}_{60}$ ) is polyhydroxylated fullerene, called fulleranol-d, obtained by direct heterogeneous-catalytic oxidation of fullerene and corresponding to the conditional formula  $\text{C}_{60}(\text{OH})_{24}$  [30]–[32]. In cathodic polarization conditions in potentiostatic or potentiodynamic modes, fulleranol ions  $[\text{C}_{60}(\text{OH})_{23}]^+$  are capable of receiving electrons by introducing a carbon film or fulleranol particles into the matrix.

To study the elemental composition of the films outer layers after cathodic treatment in the potentiostatic mode, two-layer ITO/NiO (II) films were used. Figure 5 shows an overview RF spectrum of the two-layer ITO/NiO (II) film surface layer after cathodic treatment at a potential of -800 mV for 30 s in a 2M KOH solution containing 0.2%  $\text{C}_{60}(\text{OH})_{24}$ .

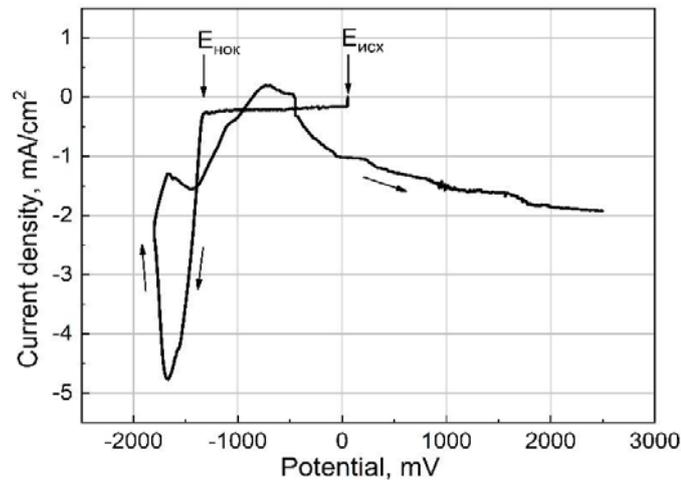


**Figure 5. Overview X-ray spectrum of a two-layer ITO/NiO film after cathodic treatment in a KOH solution containing 0.2% fulleranol  $\text{C}_{60}(\text{OH})_{24}$ .**

According to X-ray photoelectron spectroscopy, the following elements were detected in the film outer layers: Ni (3.7%), O (20.2%), C (73.8%), the rest ones to be Sn, Pt, Fe and Ca. The carbon (carbon-containing particles) proportion in the ITO film outer layers during cathodic treatment can reach about 80%.

Figure 6 shows a cyclic voltage curve obtained on ITO films in a 1M KOH solution containing 0.2%  $\text{C}_{60}(\text{OH})_{24}$ . The carbon intrusion into the ITO films outer layers during cathodic polarization does not affect the indium bronze formation. Thus, the VAC cathode branch shape, the potentials of the cathode current growth beginning and the film transition to the colored state in the presence and absence of fulleranol-d in the 1M KOH solution coincide and amount to -1.45V. Increasing the indium bronze layers conductivity made it possible to obtain the VAC anode branch. As can be seen, currents characteristic peaks are observed on it. However, the ITO film transition to a discolored state did not occur in the studied potential range. This

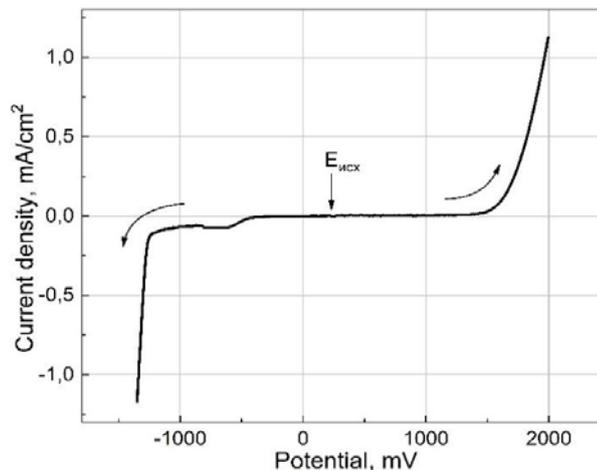
indicates that indium bronzes formed during the ITO films cathodic polarization in a 1M KOH solution containing 0.2%  $C_{60}(OH)_{24}$ , are characterized by irreversible properties and there is no electrochromic effect in them.



**Figure 6. Cyclic voltage curve obtained on ITO films in 1M KOH solution containing 0.2% fullerene  $C_{60}(OH)_{24}$ .**

Apparently, indium bronzes formed in 1M KOH solutions are characterized by a sufficiently high value of the stoichiometric coefficient  $x$  in the general formula of bronze  $H_xIn_2O_3$ . The 1M solution of KOH fullerene-d addition leads to a decrease in the transmittance of  $T_x$  ITO films in the colored state to 9.8%.

The properties of hydrogen indium bronzes formed under conditions of cathodic polarization in ITO films in a neutral 3% NaCl solution are investigated. Figure 7 shows the cathode and anode branches of the cyclic voltampere curve (VAC) obtained on an ITO film in a neutral 3% NaCl solution in the potential range from -1360 to 2500 mV.



**Figure 7. Voltamper curve cathode and anode branches obtained on ITO films in 3% NaCl solution.**

As can be seen, in a neutral 3% NaCl solution, the potential for hydrogen release beginning and ITO film staining in dark brown is observed at a more positive potential than in the 1M KOH solution and is -1.28 V (Fig. 3, curve 2). As the potential shifts in the negative direction to -1360 mV, the intensity of ITO film staining increases. The transmittance  $T_x$ , determined at a wavelength  $\lambda = 550$  nm of ITO films in the staining state at a potential  $E = -1.360$  V in a 3% NaCl solution was 6.8%, and the contrast ratio (transmittance index)  $CR = T_x/T_0$ , representing the transmittance ratio in the colored and unpainted states, relatively, was 8.12%. When the potential was deployed in the opposite anode direction, and the anode HAC branch was recorded, it was found that the colored ITO-plates state remained almost up to 1.48 V. At a potential of 1.48V, an increase in the anode current is observed on the VAC anode branch, and the ITO film moves into a discolored state.

Figure 3, Curve 2 shows (in a wide range of potentials) the dependence of ITO-strips transmittance on the potential in the processes of discoloration staining in a 3% NaCl solution. As can be seen, the

process of ITO film electrochromic staining in a 3% NaCl solution occurs in a narrow range of potentials and is characterized by asymmetry during discoloration in relation to the potential axis. The ITO film transmittance in the discoloration state at a potential of 2.0 V in a 3% NaCl solution was 64.4%, i.e. under these conditions, the ITO film is not completely discolored. Thus, the switching efficiency, denoting the proportion of electrochromic material, going from the colored state, when changing the redox process direction to the unpainted state, is 76%.

Thus, indium bronzes formed in ITO films in a neutral 3% NaCl solution are characterized by high conductivity (as well as the original ITO films) and a sufficiently small value of the stoichiometric coefficient  $x$  in the general formula of bronze  $H_xIn_2O_3$  and are capable of reversibly changing light transmission when voltage is applied, i.e. they have electrochromic properties.

## 4. Conclusions

As a result, it was found that in aqueous solutions at certain threshold potential  $E_p$ , depending on the solution pH, and more negatively, the modification of the optical and electrical properties of ITO films is observed. The properties modification is determined by the kinetics of hydrogen intercalation into the film with the formation of hydrogen-indium bronzes  $H_xIn_2O_3$ . The conductivity and electrochromic properties of the resulting bronzes are determined by the stoichiometric coefficient  $x$  value in the general formula of bronzes.

The results showed that indium bronzes formed in a neutral 3% NaCl solution with a low  $x$  index value were characterized by high conductivity and reversible dark brown coloration-discoloration of the film, i.e. had electrochromic properties. Under open circuit conditions, ITO films retained a colored state for a long time (up to a day or more), i.e. ITO films had an "optical memory effect" and were non-volatile. These bronzes could be used for premises protection from the environmental factors effects.

The results showed that indium bronzes formed in aqueous and water-ethanol 1M KOH solutions were characterized by a rather high value of the stoichiometric coefficient  $x$ , low conductivity and irreversibility of the ITO films coloring process. The increase in the bronzes conductivity due to the carbon intercalation into the ITO film did not affect the reversibility of its coloration-discoloration process.

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#### **Information about authors:**

##### **Darya Strekalovskaya**

ORCID: <https://orcid.org/0000-0001-9965-7966>

E-mail: [darya.strek@gmail.com](mailto:darya.strek@gmail.com)

##### **Ludmila Baturova**

PhD of Chemical Sciences

ORCID: <https://orcid.org/0000-0002-4740-8228>

E-mail: [lpbat@yandex.ru](mailto:lpbat@yandex.ru)

##### **Aleksandr Semench**

PhD of Chemical Sciences

ORCID: <https://orcid.org/0000-0003-2719-8726>

E-mail: [asemencha@spbstu.ru](mailto:asemencha@spbstu.ru)

##### **Elena Aleksandrova**

ORCID: <https://orcid.org/0000-0002-1143-7836>

E-mail: [aleksandrova\\_ek@spbstu.ru](mailto:aleksandrova_ek@spbstu.ru)

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