

PHOTOLYSIS OF LEAD AZIDE IN CONTACT WITH COPPER OXIDE (I)

E.P. Surovoy, L.N. Bugerko, S.V. Rasmatova

Kemerovo State University

E-mail: epsur@kemsu.ru

It is stated that alongside with decrease of photolysis velocity and photocurrent in the area of intrinsic absorption of lead azide the addition of Cu_2O extends the range of spectral sensitivity, but preliminary processing of the system by light ($\lambda=365\text{ nm}$) increases photolysis velocity. As a result of analysis of volt-ampere characteristic measurements, contact potential difference, contact photo-emf the model of system $\text{PbN}_6(\text{AM})-\text{Cu}_2\text{O}$ photolysis including stages of generation, recombination, redistribution of nonequilibrium carrier in a contact field, formation of microheterogeneous $\text{PbN}_6(\text{AM})-\text{Pb}$ systems (photolytic product), as well as formation of photolytic final products is proposed.

From the middle of the 1970's intensive development of heterogeneous system research «light-sensitive salt – metal (semiconductor)» has been stimulated by scientific society aiming at development of methods and means of more rational using of those resources which are left on our planet [1, 2]. One can point out the two main reasons for the research interest in the given field. Firstly, to provide many processes the light of sun range is used, but in some practically important photostimulated reaction the utilization of energy achieves several tens of percents. Secondly, the important applied tendencies of research are: development of fundamentally new materials for production of thermoregulating and heat-reflecting coating permitting to save up to 70 % of thermal energy, colouring agents in colouring compositions, elements of semiconductor devices etc., to record, store, and transform information transmitted by heat, optical, mechanical, and other methods [1–7].

The study of dark and photoprocesses in heterosystems on the bases of lead azide [3, 8–12], one of the components of which is lead azide combining the advantages of model compounds (has relatively uncomplicated composition and structure, simple composition of final photodecomposition products, possesses sufficient photosensitivity and essential internal photoelectric effect) is used in technology and urgent in both scientific and practical respect.

In the present paper the results of work directed to investigation of affect of copper oxide (1) additive on kinetic and spectral regularities of lead azide at pressure $1 \cdot 10^{-5}$ Pa and to determination of reasons causing by Cu_2O additive the observed changes of photochemical and photoelectric sensitivity of lead azide.

Objects and methods of research

Lead azide (AM type) – $\text{PbN}_6(\text{AM})$ was synthesized by the method of double jet crystallization by slow (during 60 min) pouring «jet in jet» aqueous 0,2n solutions of twice recrystallized commercial sodium azide and lead nitrate (ch.p. qualification) at pH 3. Samples for investigation had been prepared by careful mixing (in dry state and in ethyl alcohol) of corresponding portions of lead azide and copper oxide (1) with subsequent drying and pressing at pressure $4 \cdot 10^3 \text{ kg}\cdot\text{sm}^{-2}$ of tablets of 1 sm diameter. Besides, copper oxide (1) was applied by the method of thermal evaporation at pressure $1 \cdot 10^{-3}$ Pa us-

ing vacuum universal post ВУП-5М on illuminated surface of $\text{PbN}_6(\text{AM})$ tablets. Comparing the results and drawing the curves of spectral distribution of photolysis velocity (V_{ph}), photocurrent (i_{ph}) and photo-emf (U_{ph}) light transmission through Cu_2O was taken into account. V_{ph} , i_{ph} and U_{ph} of samples were measured at pressure $\sim 10^{-5}$ Pa. Measuring V_{ph} lamp PMO-4C of omega-tron mass-spectrometer ИПДО-1 tuned in the frequency of molecular nitrogen registration was used as a sensor [13]. Changes of i_{ph} and U_{ph} were carried out in the device including electrometric voltmeter B7-30 [12]. Spectrums of diffuse reflection (DR) were measured before and after illumination of samples at pressure $\sim 10^{-4}$ Pa using the device [14], by spectrophotometer СФ-4А with attachment ПДО-1 and at pressure 101,3 kPa by spectrophotometer Specord-M40 with attachment for reflection $8^\circ/d$ [15]. As sources of illumination mercury (ДРТ-250) and xenon (ДКсШ-1000) lamps were used. To distinguish a necessary section of spectrum monochromator МДР-2 and СІМ-2, light filters were used. Actinometry of light sources was performed by means of radiation thermocouple PT-0589. Contact potential difference between $\text{PbN}_6(\text{AM})$, Cu_2O and comparison platinum electrode was measured by means of modified Kelvin's method [9]. Topography of solid-phase photolysis products $\text{PbN}_6(\text{AM})$ and $\text{PbN}_6(\text{AM})-\text{Cu}_2\text{O}$ was studied by the method of carbon replicas with extraction by transmission microscope УЭМБ-1000.

Results and discussions

As a result of comparison of kinetic curves V_{ph} (fig. 1), changed by the action of light from the region of intrinsic absorption of lead azide on the samples $\text{PbN}_6(\text{AM})$ and $\text{PbN}_6(\text{AM})-\text{Cu}_2\text{O}$ it was stated that system creation along with decrease of additive Cu_2O V_{ph} $\text{PbN}_6(\text{AM})$ does not result in change of form of kinetic curves.

In the fields of intensive illumination ($I > 1 \cdot 10^{14} \text{ quant}\cdot\text{sm}^{-2}\cdot\text{s}^{-1}$) of systems $\text{PbN}_6(\text{AM})-\text{Cu}_2\text{O}$ the characteristic for individual lead azide [13] sections appear: I – non-stationary, II – stationary, III – growth, IV – saturation. Decrease of falling light intensity ($I < 1 \cdot 10^{14} \text{ quant}\cdot\text{sm}^{-2}\cdot\text{s}^{-1}$) results in decrease of V_{ph} , as well as increase of length of kinetic curve sections V_{ph} . At large concentrations of additives (more than 30 %) darkening (screening) of surface part of lead azide takes place.

Long (more than one month) keeping of the systems involved in the «atmospheric» conditions, preliminary heat and light treatments as well as treatment of lead azide (before system creation) in the reducing medium decrease (up to disappearing) the initial maximum on the kinetic curves V_{ph} . As an example in fig. 1 (curves 2–4) the research results of influence of preliminary treatment of samples $PbN_6(AM)-Cu_2O$ by the light from the region of lead azide intrinsic absorption. It is seen (fig. 1, curve 3) that repeated (after light interruption on section II) illumination of samples does not result in significant change of V_{ph} values in II, III and IV sections of V_{ph} kinetic curves. After preliminary light treatment of samples up to the IV section of V_{ph} it monotonously increases up to constant value and corresponds to the values of V_{ph} in section IV of not-light-treated samples (fig. 1, curve 4). Longer illumination of samples results in decrease of V_{ph} . As a result of electron-microscopy and spectrophotometric examination it was stated that the decrease of V_{ph} observed is connected with darkening of sample surface by metal (product of photolysis) and, hence, with decrease of the number of light quanta absorbed by the system [13]. Subsequent treatment of preliminary exposed systems in oxidizing medium, keeping in «atmospheric» conditions and at pressure $1 \cdot 10^{-1}$ Pa during one month result in partial reduction of V_{ph} kinetic curve form.

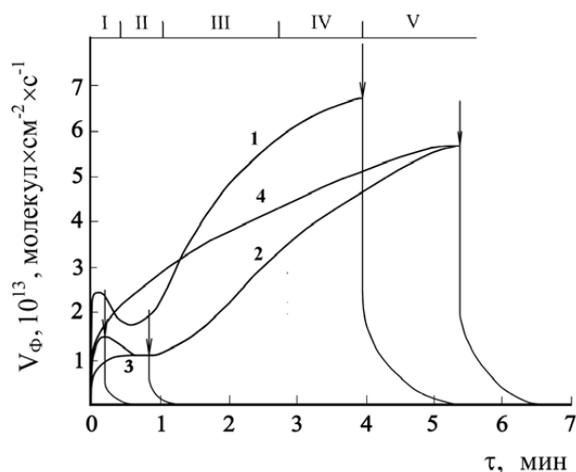


Fig. 1. Kinetic curves of photolysis velocity $PbN_6(AM)$ (1), systems $PbN_6(AM)-Cu_2O$ (2–4) before (1,2) and after light interruption for systems $PbN_6(AM)-Cu_2O$ in II (3), IV (4) sections. $I=2 \cdot 10^{15}$ quant \cdot sm $^{-2}$ \cdot s $^{-1}$, $\lambda=365$ nm. The moments of light interruption are denoted by arrows

After cease of sample exposure in different sections of V_{ph} kinetic curves the section of post-gas-evolution (V) is observed (fig. 1). It is seen that the curves of post-gas-evolution consist of the two sections – «quick» and «slow». With increase of exposure time and intensity of falling light duration of post-gas-evolution increases due to increase of time interval of «slow» constituent, but with decrease of temperature the section of post-gas-evolution reduces owing to decrease of time interval of «slow» constituent. It is stated that at 293 K anamorphosis of post-gas-evolution for $PbN_6(AM)-Cu_2O$ constructed in coordinates $\ln C_{N_2} = f(\tau)$, independently on time of preliminary exposure, intensity of falling light are linear. In table 1 velocity constants (k) of the

process responsible for post-gas-evolution, calculated by kinetic curves are presented after cease of illumination in I, II, and IV sections of V_{ph} kinetic curves of the samples involved.

Table 1. Velocity constants (k , s $^{-1}$) of the process responsible for post-gas-evolution (section V) after light interruption in I, II and IV sections of V_{ph} kinetic curve

Sample	I	II	IV
$PbN_6(AM)-Cu_2O$	$(4,40 \pm 0,05) \cdot 10^{-2}$	$(4,20 \pm 0,02) \cdot 10^{-2}$	$(2,40 \pm 0,02) \cdot 10^{-3}$

In fig. 2 V_{ph} spectral distributions of $PbN_6(AM)-Cu_2O$ systems at illumination of them by the light if equal intensity at 293 K constructed in stationary sections (II) of V_{ph} kinetic curves. It is seen that creation of $PbN_6(AM)-Cu_2O$ systems along with decrease of V_{ph} in the region of $PbN_6(AM)$ intrinsic absorption results in noticeable photodecomposition in the long-wave region of spectrum corresponding to the region of absorption and photoelectric sensitivity of Cu_2O . To find out the reasons producing the observed changes by Cu_2O V_{ph} $PbN_6(AM)$ additive a series of volt-ampere characteristics (VAC) measurements of i_{ph} and U_{ph} was performed. As a result of VAC measurements in the range of external voltage $-3 \dots +3$ V it was stated that the effect of «rectification» is absent. In spectrum regions corresponding to absorption regions and photoelectric sensitivity of contact partners' noticeable i_{ph} and U_{ph} (fig. 2) were found. It is seen that curves of spectral distribution of U_{ph} , V_{ph} and i_{ph} correlate, but the sign U_{ph} from the negative side of $PbN_6(AM)$.

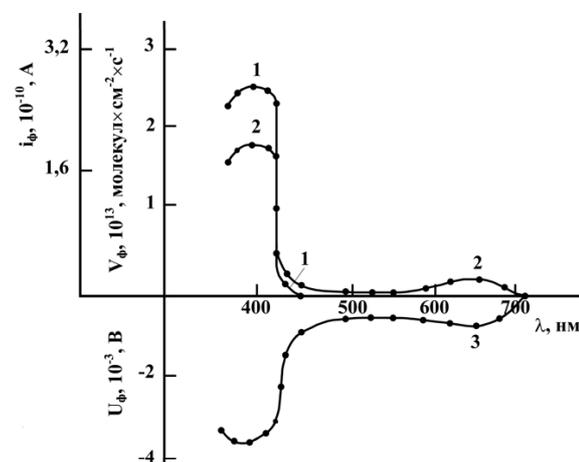


Fig. 2. Spectral distributions of photolysis velocity (1, 2), photocurrent (1, 2) and photo-emf (3) of $PbN_6(AM)$ (1), $PbN_6(AM)-Cu_2O$ (2,3) systems. $I=3,17 \cdot 10^{15}$ quant \cdot sm $^{-2}$ \cdot s $^{-1}$

Regularities of solid-phase photolysis product formation of $PbN_6(AM)-Cu_2O$ system was studied by means of DR measurement of samples before, in the process, and after treatment by the light from the region of intrinsic absorption of lead azide in the range of falling light intensity $7,95 \cdot 10^{14} \dots 5,56 \cdot 10^{15}$ quant \cdot sm $^{-2}$ \cdot s $^{-1}$. Long-wave edge of $PbN_6(AM)$ [13] and $PbN_6(AM)-Cu_2O$ DR amounts $\lambda=400$ nm (fig. 3). At the times of illumination corresponding to realization of I and II sections on V_{ph} kinetic curves along with DR decrease in the range of 400...800 nm on DR spectral curves the maximum appe-

ars at $\lambda \approx 400 \dots 450$ nm. Further increase of time of light treatment (up to the IV section) results in displacement of maximum into long-wave region of spectrum. Keeping of illuminated samples during 24 h at 293 K and $P=101,3$ kPa results in partial regeneration of DR samples in the region $\lambda \geq 400$ nm. Kinetic curves of V_{ph} concentration change dependencies of photolytic metal (C_{me}) were constructed by calculating the results of changes at different intensivities of falling light with square values (S), corresponding to DR changes of $PbN_6(AM)-u_2O$ systems in the process of illumination. The found out coincidence of dependencies as well as the results presented in [13] show that changes observed as a result of sample illumination on DR curves of $PbN_6(AM)-Cu_2O$ systems are explained by lead formation (lead azide photolysis product). Solid-phase (lead) and gas (nitrogen) products of $PbN_6(AM)-Cu_2O$ system photolysis are formed in stoichiometric relation, mainly on the sample surface. In table 2 $PbN_6(AM) V_{ph}$ and $PbN_6(AM)-Cd$ system constants estimated by tangent of dependency slope angle $\ln S=f(\tau)$, $\ln C_{me}=f(\tau)$ are presented. From table 2 it is seen that V_{ph} constants of lead azide and $PbN_6(AM)-Cd$ systems virtually coincide.

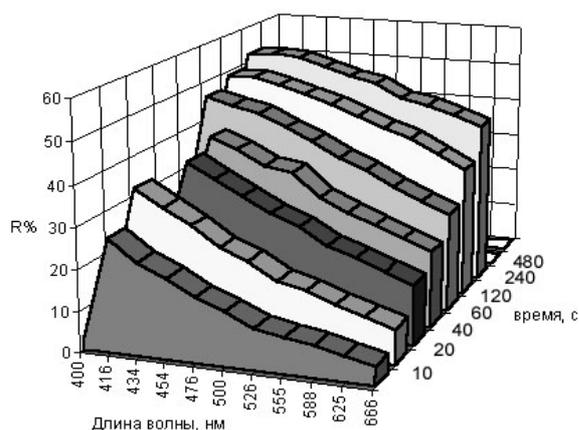


Fig. 3 Changes of reflective ability of $PbN_6(AM)-Cu_2O$ systems, depending on time of light illumination: 1) 10, 2) 20, 3) 40, 4) 60, 5) 120, 6) 240, 7) 480 s. $\lambda=365$ nm, $I=3,17 \cdot 10^{15}$ quant \cdot sm $^{-2} \cdot$ s $^{-1}$

Table 2. Constants of photolysis velocity calculated by kinetic curves of photolysis velocity (k_{1ph}) and spectra of diffusive reflection (k_{1DR}), $I=3,17 \cdot 10^{15}$ quant \cdot sm $^{-2} \cdot$ s $^{-1}$

Sample	k_{1ph}, s^{-1}	k_{1DR}, s^{-1}
$PbN_6(AM)$	$(5,90 \pm 0,47) \cdot 10^{-2}$	$(5,80 \pm 0,48) \cdot 10^{-2}$
$PbN_6(AM)-Cu_2O$	$(5,80 \pm 0,50) \cdot 10^{-2}$	$(5,30 \pm 0,48) \cdot 10^{-2}$

The results presented in this work and obtained before [4–10] indicate the contact, photoelectric nature of change effects by additive $Cu_2O V_{ph}$ of lead azide observed in different spectral regions. It, first of all, follows from the facts revealed by the experiment:

- correlation of V_{ph} , i_{ph} and U_{ph} spectral distribution curves;
- correlation of V_{ph} , i_{ph} and U_{ph} spectral distribution curves with absorption spectra and i_{ph} $PbN_6(AM)$ and Cu_2O spectral distribution curves;

- formation of U_{ph} indicates directly division of non-equilibrium charge carriers on the contact at illumination.

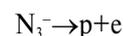
Photochemical evidences of photoelectric processes in $PbN_6(AM)-Cu_2O$ systems can be produced by redistribution under the action of electron – hole couple contact field, generated by light in the region of spatial charge (RSC) of contacting partners which results in Cu_2O additive displaying as a donor or acceptor of non-equilibrium charge carrier with respect to lead azide. According to relations of thermoelectric work of contact partners output (table. 3) [9], the effect of «rectification» on VAC as well as the same over the whole spectrum, but different (depending on experiment conditions) sign of U_{ph} should be expected.

Table 3. Contact difference of potentials (B) between $PbN_6(AM)$, Cu_2O and relative platinum electrode at 293 K

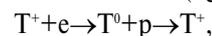
Preparation	$P=1 \cdot 10^5$ Pa	$P=1 \cdot 10^{-5}$ Pa
$PbN_6(AM)$	+0,28	+0,46
Cu_2O	+0,31	+0,20

However, as the examination showed that noticeable «rectification» effects are absent on VAC independently on relations of thermoelectric work of contact partners output but sign U_{ph} from the direction of $PbN_6(AM)$ measured in atmospheric conditions and in vacuum for $PbN_6(AM)-Cu_2O$ systems is negative (fig. 2). The facts mentioned as well as the results of potential contact difference measurements (table 3) [9], capacitor photo-emf [11], V_{ph} , i_{ph} and U_{ph} of $PbN_6(AM)$ -metal (photolysis product) system [13] point to the fact of significant concentration and crucial role of intrinsic surface electron states (T_{π}^{-} , T_{κ}^{+}) of lead azide and surface electron states of contact (Π_{π}^{-} , Π_{κ}^{+}) of lead azide with copper oxide (1) in the processes of redistribution of charge carrier on the contacts in darkness and at their illumination. Producing contacts of $PbN_6(AM)$ with Cu_2O the process of exchange of equilibrium charge carrier takes place up to the moment of setting up thermodynamic equilibrium. The character of surface bends of zones near $PbN_6(AM)$ and Cu_2O contacting with it, determining the type of donor-acceptor affect of semiconductor on $PbN_6(AM)$ photolysis, is presented in fig. 4.

Under the action of light from the region of intrinsic absorption of lead azide on $PbN_6(AM)-Cu_2O$ systems the intensive generation of electron-hole couples in lead azide and in semiconductor takes place (fig. 4, transitions 1, 2).



As quant output of $PbN_6(AM)-Cu_2O$ system photolysis $\tau \approx 60$ s 0,002...0,010 at exposure, the part of generated charge carriers recombines (fig. 4, transition 3)



where T^{+} – recombination centre. Couples of nonequilibrium charge carriers of lead azide and copper oxide (1) generated in RSC are redistributed in contact field formed owing to discrepancy of work of contact partners output (table 3), presence of T_{π}^{-} , T_{κ}^{+} and Π_{π}^{-} , Π_{κ}^{+} . Nonequilibrium holes of lead azide valent zone and

nonequilibrium electrons of Cu_2O conductivity zone move to T_{Π}^- , T_K^+ level.

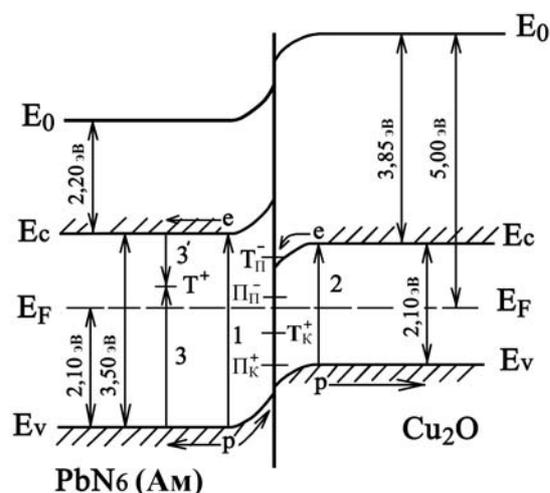


Fig. 4. Diagram of energy bands of $\text{PbN}_6(\text{AM})-\text{Cu}_2\text{O}$ system. E_v – level of valent zone, E_c – level of conductivity zone bottom, E_f – Fermi level, E_0 – vacuum level, T^+ – recombination centre

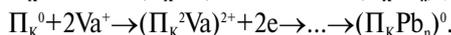
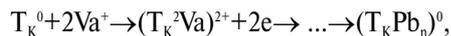
Electrons and holes settled on Π_{Π}^- , Π_K^+ levels can recombine or exchange with the nearest zones of semiconductor and lead azide.

At exposure of $\text{PbN}_6(\text{AM})-\text{Cu}_2\text{O}$ systems by light from the region of Cu_2O absorption intensive generation of electron-hole couples in semiconductor takes place (fig. 4, transition 2). Nonequilibrium Cu_2O charge carriers generated in RSC redistribute in contact field with moving of electrons from conductivity zone of semiconductor into T_{Π}^- , T_K^+ and Π_{Π}^- , Π_K^+ levels. The realized sign U_{ph} (fig. 2) from the lead azide direction for $\text{PbN}_6(\text{AM})-\text{Cu}_2\text{O}$ systems indicates the possibility to perform the transitions involved. Simultaneously with the mentioned transitions, which result in U_{ph} formation (and displacement of energy levels of contacting partners) the flows of equilibrium charge carriers take place. As a result, hole concentration in RSC of lead azide (in contact with Cu_2O) will change in comparison with their concentration in individual azide. Resultant change of hole concentration in RSC of lead azide results in corresponding decrease of i_{ph} and V_{ph} in intrinsic region of azide absorption (fig. 1, 2) and appearance of i_{ph} and photolysis in long-wave region of spectrum (fig. 2), corresponding region of absorption and photoelectric sensitivity of Cu_2O by taken reactions of nitrogen formation for heavy metal azide photolysis:

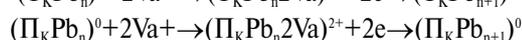


where V_K^- и V_A^+ – cation and anion vacancies.

We suppose that T_{Π}^- , T_K^+ of lead azide and Π_{Π}^- , Π_K^+ are centres of photolytic metal with participation of moving anion vacancies (lead azide is disordered by Schottky [16])



The decrease of V_{ph} observed at the initial section of kinetic curve in the process and after preliminary exposure of samples (fig. 1) confirms irreversible flow rate of surface centres. In the process of growth of photolytic metal particles microheterogeneous «lead azide – lead (photolysis product)» systems are formed [13]. Under the action of light from region of intrinsic absorption of lead azide couples of carriers generated in RSC of lead azide are redistributed in contact field formed due to discrepancy between thermoelectric works of lead azide output and photolytic lead because of transition of nonequilibrium holes of $\text{PbN}_6(\text{AM})$ valent zone into lead. Simultaneously, photoemission of electrons from lead into conductivity zone of lead azide takes place. These processes can stimulate diffusion of anion vacancies to the growing particles.



We suppose that at photolysis of $\text{PbN}_6(\text{AM})-\text{Cu}_2\text{O}$ heterosystems (as well as for $\text{PbN}_6(\text{AM})$ [13]) the increase of not only sizes, but also the number of photolytic lead particles takes place [12]. As a result hole concentration in RSC of lead azide and V_{ph} (fig. 1, section III) grows. In the process of photolysis the $\text{PbN}_6(\text{AM})-\text{Cu}_2\text{O}$ contact boundaries is covered by the layer of photolytic lead and at more degree of transformation photochemical processes in $\text{PbN}_6(\text{AM})-\text{Cu}_2\text{O}$ systems is significantly defined by photoelectric processes on azide – lead (photolysis product) – semiconductor boundary. To define the limiting stage of growth process of photolytic lead particles time during which moving anion vacancy neutralizes electron or diffuses towards neutral centre. Relaxation time according to drift mechanism is equal to Maxwell's relaxation time [17]

$$\tau_i = \varepsilon / 4\pi\sigma,$$

where ε – permittivity ($\varepsilon_{\text{PbN}_6} = 6$), σ – specific conductivity at 293 K ($\sigma_{\text{PbN}_6} \approx 1 \cdot 10^{-12} \text{ Om}^{-1} \cdot \text{sm}^{-1}$), $\tau_i = 0,4 \text{ s}$. Constant of photolysis velocity in this case amounts $k^I = 2,5 \text{ s}^{-1}$.

Relaxation time at diffusion process can be estimated as [17]

$$\tau_d = e^2 / \sigma k_b a T,$$

where e – electron charge; a – lattice constant ($a_{\text{PbN}_6} = 8 \cdot 10^{-10} \text{ sm}$); $T = 293 \text{ K}$, k_b – Boltzmann constant. At 293 K $\tau_d = 80 \text{ s}$. Constant of photolysis velocity (k^{II}) in this case comes to $k^{II} \approx 1,25 \cdot 10^{-2} \text{ s}^{-1}$.

Satisfactory fit of constants of photolysis velocity (table 2) with k^{II} give grounds to suggest that limiting stage of photolysis process of $\text{PbN}_6(\text{AM})-\text{Cu}_2\text{O}$ is diffusion of anion vacancies towards neutral centre.

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RESEARCH OF ACIDITY OF THE SURFACE OF POWDERS AND PROPERTIES OF THIN FILMS OF $\text{Ta}_2\text{O}_5 - \text{La}_2\text{O}_3$ SYSTEM, OBTAINED BY SOL-GEL METHOD

V.V. Kozik, O.V. Liseenko, L.F. Ikonnikova, L.P. Borilo

Tomsk state university
E-mail: borilo@mail.ru

Thin films and powders of $\text{Ta}_2\text{O}_5 - \text{La}_2\text{O}_3$ systems are obtained by sol-gel technology. The physical and chemical properties of the synthesized films: adhesion, electric resistance, thickness, coefficient of refraction and acidic-basic properties of the powder surface are investigated. Diagrams of the condition «composition – coefficient of refraction, surface acidity» are built.

Research and regulation of physical and chemical surface properties of solids obtained by sol-gel method, as well as studying of depth and direction of the processes proceeding with participation of a formed solids phase, is the important problem of applied chemistry. Stability of fastening of films on a surface, optical and physical properties of films define possibility of their practical use. The acidic-basic properties are universal physical and chemical criterion of a solids surface which depends on chemical nature of substance, way of its preparation, chemical composition of system and quantity of impurity on a surface, therefore studying of these characteristics is the actual problem for thin films, too. In this connection these properties of thin films and disperse powders of the $\text{Ta}_2\text{O}_5 - \text{La}_2\text{O}_3$ system obtained by sol-gel method were studied in this work.

Experimental part

Samples of the $\text{Ta}_2\text{O}_5 - \text{La}_2\text{O}_3$ system with La_2O_3 contents from 0 up to 100 mol. % have been obtained by sol-gel technology from the film-forming solutions [1]. The film-forming solutions have been prepared by dissolution TaCl_5 and $\text{LaCl}_3 \cdot 7\text{H}_2\text{O}$ in dried ethanol. The films have been obtained on the MPW-340 centrifuge with rotation speed 2500 rot./min. As substrates one used single crystalline silicon of KЭФ-10 mark. Thermal processing of the films after preliminary drying at 333 K was carried out in a muffle furnace at temperature 873 K. Composition of films and powders was defined with the ДРОН-3М diffractometer, CuK_α -radiation ($\lambda=1,5418$ nm); Ni-filter. Adhesion of films to a substrate was measured with the ПИМТ-3 hardness microtester. Index of refraction and thickness of oxide films