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PHOTOCHEMISTRY AND MAGNETOCHEMISTRY

Rules Governing the Formation of Photolysis Products in the Lead Azide–Copper(I) Oxide System

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Abstract—It was found that, along with a decrease in the rate of photolysis and photocurrent in the region of lead azide intrinsic absorption, the addition of copper(I) oxide broadened the range of spectral sensitivity, and preliminary treatment of the PbN_6(Ab)–Cu_2O system with light ($\lambda = 365$ nm) increased the rate of photolysis. The rate constants for photolysis were estimated. An analysis of the results of current-voltage characteristic, contact potential difference, and contact photo-electromotive force measurements was used to construct a diagram of energy zones and suggest a model of the photolysis of the PbN_6(Ab)–Cu_2O system including stages of the generation, recombination, and redistribution of nonequilibrium carriers in a contact field, formation of microheterogeneous PbN_6(Ab)–Pb (photolysis product) systems, and formation of final photolysis products.

Keywords: photolysis, lead azide–copper(I) oxide system, diagram of energy zones
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INTRODUCTION

Studies of the nature and rules governing processes in light sensitive salt–metal (semiconductor) heterogeneous systems are of interest both for solid state physics and chemistry and general heterogeneous catalysis theory and in relation to the currently developing possibility of creating new systems on their basis with a controlled level of photosensitivity [1–7]. This makes it necessary, along with studies of the photochemical decomposition of inorganic compounds that offer promise for use in practice, to extend works aimed at changing their photosensitivity with the purpose of determining the general rules governing this phenomenon for a wide range of objects and formulating particular recommendations for controlling photochemical sensitivity of various materials [1–3]. Studies of dark and photo processes in heterosystems based on lead azide [1, 8–12], having qualities of model compounds (comparatively simple composition and structure, fairly substantial photochemical sensitivity, photolysis products (lead and nitrogen) not interacting with each other, substantial internal photoelectric effect) and is used in technology, are of current interest for both science and practical applications.

In this work, we report the results of studies of the kinetic and spectral rules governing the formation of photolysis products in the lead oxide–copper(I) oxide heterosystem before and after preliminary light treatment in a vacuum (1×10^{-5} Pa). Our purpose was to determine the reasons that caused changes in photochemical and photoelectric sensitivity of lead azide observed when Cu_2O was added.

EXPERIMENTAL

Lead azide of Ab grade (PbN_6(Ab)) was synthesized by two-stream crystallization, by simultaneously pouring together aqueous 0.2 N solutions of doubly recrystallized commercial sodium azide and lead nitrate of kh. ch. (chemically pure) grade at pH 3 and $T = 293$ K during 1–2 s [8–10]. Samples were prepared by thoroughly mixing (in the dry state and in ethanol) certain amounts of PbN_6(Ab) and copper(I) oxide followed by drying and pressing at a 1×10^3 kg/cm^2 pressure into pellets 0.5–1 cm in diameter. In addition, copper(I) oxide was deposited on an illuminated surface of PbN_6(Ab) pellets by thermal vaporization in a vacuum (1×10^{-3} Pa) using a VUP-5M universal vacuum unit. In comparing the results and ploting curves of the spectral distribution of the rate of photolysis (V_p), photocurrent (i_p), and photoelectromotive force (photo-EMF, U_p), light transmission through Cu_2O was taken into account.

Sample V_p, i_p, and U_p measurements were performed on experimental complexes that provided high-vacuum conditions (1×10^{-5} Pa). The source of radiation was mercury (DRT-250) and xenon (DKsSh-1000) lamps. The required spectral regions were isolated using MDR-2 and MSD-1 monochromators and a set of light filters. Actinometry of light sources was performed using an RT-0589 radiation

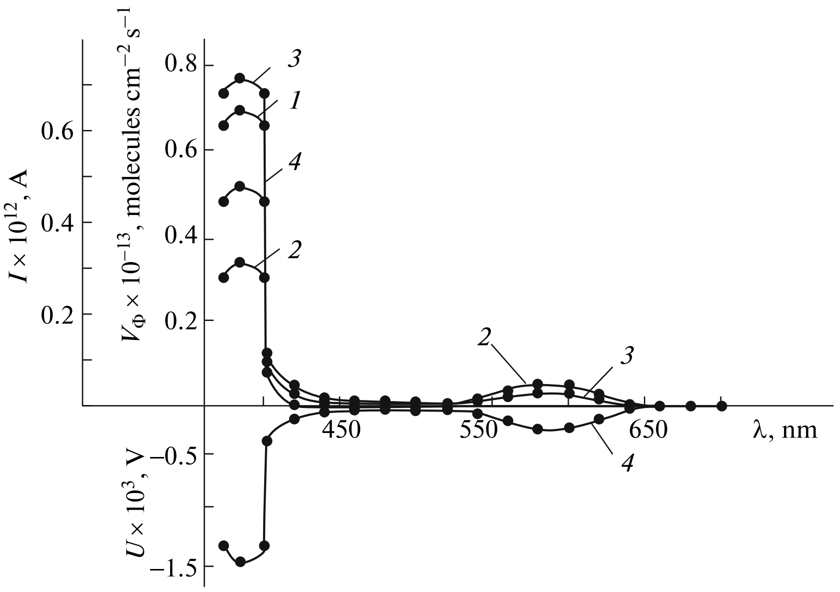

Fig. 1. Spectral distributions of (*1*, *2*) photolysis rate, (*3*, *4*) photocurrent, and (*5*) photo-EMF; (*1*, *3*) PbN_6(Ab) and (*2*, *4*, *5*) PbN_6(Ab)–Cu_2O; $I = 2 \times 10^{15}$ quanta cm^{-2} s^{-1}.

thermoelement. In V_p measurements, we used an RMO-4S tube of IPDO-1 omegatron mass spectrometer tuned to the frequency of recording molecular nitrogen [11] as a sensor. Measurements of i_p and U_p were performed using a V7-30 electrometric voltmeter or a TR-1501 electrometer [12]. Diffuse reflectance spectra before and after sample irradiation were measured at a pressure of 101.3 kPa on a Specord-M40 spectrophotometer using an 8^0d accessory for reflection [13] and in a vacuum (1×10^{-4} Pa) using a device described in [14]. The contact potential difference (CPD) between PbN_6(Ab), Cu_2O, and a platinum reference electrode was measured using a modified Kelvin method [15]. The topography of solid-state PbN_6(Ab) and PbN_6(Ab)–Cu_2O photolysis products was studied using extractive carbon replicas on a UEMV-1000 microscope [8].

RESULTS AND DISCUSSION

In an analysis of the kinetic and spectral characteristics of the photolysis of PbN_6(Ab) and the PbN_6(Ab)–Cu_2O system, we found that along with a decrease in V_p in the region of intrinsic absorption of PbN_6(Ab) caused by the addition of copper(I) oxide, V_p spectral distribution curves constructed using stationary regions (II) of V_p kinetic curves contained a new long-wave region of spectral sensitivity corresponding to the region of absorption and photoelectric sensitivity of Cu_2O (Fig. 1). In fields of intense ($I > 1 \times 10^{14}$ quanta cm^{-2} s^{-1}) irradiation of the PbN_6(Ab)–Cu_2O system by light in the region of lead azide intrinsic absorption edge (λ = 380 nm), regions characteristic of PbN_6(Ab) [8–10] appeared on V_p kinetic curves, nonstationary (I), stationary (II), increase (III), and saturation (IV) (Fig. 2).

As the intensity of incident light decreased ($I < 1 \times 10^{14}$ quanta cm^{-2} s^{-1}), we observed V_p lowering and extension of V_p kinetic curve regions. At high (more than 30%) admixture concentrations, we observed shading (screening) of part of lead azide surface and, as a consequence, a decrease in V_p. Long-term (for more than a month) storage of the systems studied under "atmospheric" conditions, preliminary thermal and light treatments, and treatment of lead azide in a reducing medium (before system creation) decreased (up to complete disappearance) the initial V_p curve maximum. By way of example, the results of studies of the influence of preliminary treatment of PbN_6(Ab)–Cu_2O samples by light in the region of intrinsic absorption of lead azide are shown in Fig. 2. We see (Fig. 2, curves *2*, *3*) that repeated (after radiation interruption in regions I and II) irradiation of samples does not cause noticeable changes in V_p in regions II, III, and IV of V_p kinetic curves. After preliminary light treatment of samples to region IV, the V_p value monotonically increases to a constant value and corresponds to V_p in region IV of samples not subjected to light treatment (Fig. 2, curves *1*, *4*). More prolonged sample illumination decreases V_p. Electron microscopic and spectrophotometric studies showed that the observed decrease in V_p was related to shading of the surface of samples by a solid-state photolysis product and, as a consequence, a decrease in the number of light quanta absorbed by the system [8–10]. Subsequent treatment of preliminarily exposed systems in an oxidizing medium and storage under atmospheric conditions and in a vacuum (1×10^{-1} Pa) for a month

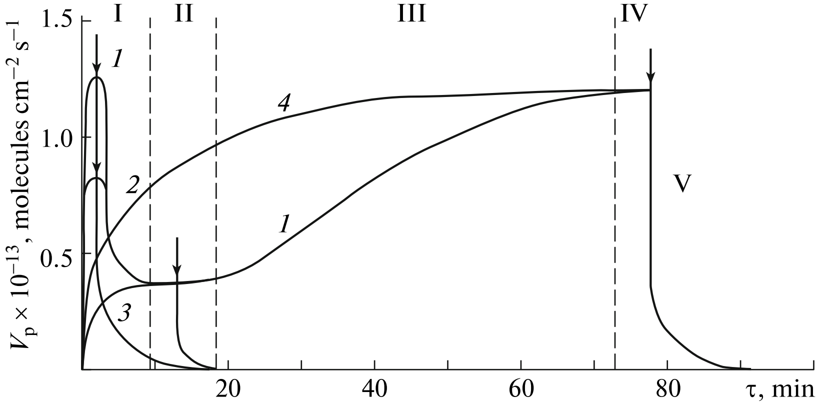

Fig. 2. Kinetic curves of the rate of photolysis of the PbN_6(Ab)–Cu_2O system at λ = 380 nm and $I = 2 \times 10^{15}$ quanta cm^{-2} s^{-1} (*1*) before and after irradiation interruption in regions (*2*) I, (*3*) II, and (*4*) IV. Arrows indicate moments of switching light off.

caused partial restoration of the form of V_p kinetic curves.

After irradiation of the PbN_6(Ab)–Cu_2O system stopped in early V_p kinetic curve regions, we observed region V of post gas release (Fig. 2). We see that post gas release curves consist of two regions, "fast" and "slow." As the time of exposure and incident light intensity increase, the duration of post gas release grows because of an increase in the duration of the slow stage. Post gas release anamorphoses constructed for PbN_6(Ab)–Cu_2O at $T = 293$ K in the $\ln c_{N_2} = f(\tau)$ coordinates are linear irrespective of preliminary exposure duration and incident light intensity. Table 1 contains rate constants (k) for the process responsible for post gas release.

The rules governing the formation of the solid state product of the photolysis of the PbN_6(Ab)–Cu_2O system were studied by diffuse reflectance spectrum measurements before, during, and after light treatment in the region of intrinsic absorption of lead azide at incident light intensities of 7.95×10^{14}–5.56×10^{15} quanta cm^{-2} s^{-1}. The long-wave edge of diffuse reflectance of PbN_6(Ab) [8–10] and PbN_6(Ab)–Cu_2O was λ = 410 nm (Fig. 3). The formation of PbN_6(Ab)–Cu_2O mixtures and their treatment with light at λ = 380 nm decreased diffuse reflectance of PbN_6(Ab) over the wavelength range 410–850 nm. At irradiation times corresponding to regions I and II of V_p kinetic curves, reflectance bands with maxima at $\lambda \approx 470$ and 600 nm appeared in diffuse reflectance spectra. An increase in the duration of light treatment to region IV caused broadening of bands and shifted maxima to longer waves. Storage of irradiated samples for 24 h at $T = 293$ K and $p = 101.3$ kPa partly restored the diffuse reflectance spectra of samples at $\lambda \geq 410$ nm.

We compared the kinetic curves of the dependences of changes in the content of photolytic metal (c_{Me}) calculated from the results of V_p measurements at various incident light intensities with the areas (S) corresponding to changes in diffuse reflectance spectra of the PbN_6(Ab)–Cu_2O system during irradiation (Fig. 4). The coincidence of the dependences and the results obtained in [8–10] show that changes in the diffuse reflectance spectra of the PbN_6(Ab)–Cu_2O system observed as a result of sample irradiation are caused by the formation of lead (photolysis product), and maxima are caused by the formation of lead particles with certain sizes. The solid state (lead) and gas phase (nitrogen) PbN_6(Ab)–Cu_2O system photolysis products are formed in the stoichiometric ratio and, largely, on the surface of samples. Table 2 contains rate constants for the photolysis of PbN_6(Ab)–Cu_2O estimated from the slope of the $\ln S = f(\tau)$ and $\ln c_{Me} = f(\tau)$ dependences. It follows from Table 2 that the V_p constants of lead azide and the PbN_6(Ab)–Cu_2O system almost coincide.

To determine the energy structure of PbN_6(Ab)–Cu_2O contacts and the reasons for the observed changes in V_p(PbN_6(Ab)) in various spectral regions caused by the addition of Cu_2O, we performed measurements of current-voltage characteristics, i_p, and U_p of the PbN_6(Ab)–Cu_2O system. We also measured contact potential differences between PbN_6(Ab), Cu_2O, and platinum electrode (Table 3). Measurements of current-voltage characteristics at external voltages of from –3 to +3 V showed that there was no "rectification" effect. Noticeable i_p and U_p were observed in the spectral regions corresponding to the regions of absorption and photoelectric sensitivity of

Table 1. Rate constants ($k \times 10^2$, s^{-1}) for the process responsible for post gas release (region V) after illumination interruption in the I, II, and IV kinetic curve V_p regions

Sample	I	II	IV
PbN_6(Ab)	3.91 ± 0.11	2.46 ± 0.01	0.270 ± 0.015
PbN_6(Ab)–Cu_2O	(3.20 ± 0.13) × 10^{-2}	3.00 ± 0.09	0.32 ± 0.05

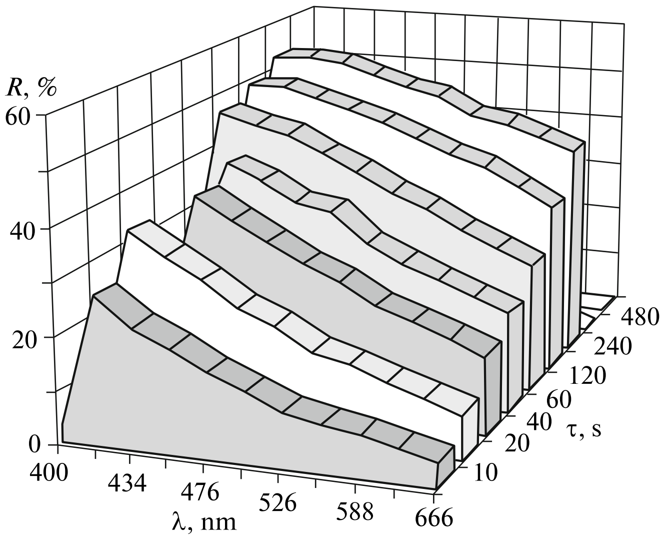

Fig. 3. Changes in the reflection ability of the $PbN_6(Ab)$–Cu_2O system depending on the duration of irradiation at $\lambda = 380$ nm, $I = 3.17 \times 10^{15}$ quanta cm^{-2} s^{-1}.

the contacting partners (Fig. 1). We see that the U_p, V_p, and i_p spectral distribution curves correlate, and the sign of U_p is negative on the side of $PbN_6(Ab)$.

The results of studies of dark and photo processes in lead azide and systems based on it presented in this work and [8–10] are evidence of the contact photoelectric nature of the observed changes in V_p of lead azide in various spectral regions as Cu_2O is added. This follows from the observations according to which

(1) V_p, i_p, and U_p spectral distribution curves of the $PbN_6(Ab)$–Cu_2O system correlate,

(2) V_p, i_p, and U_p spectral distribution curves of the $PbN_6(Ab)$–Cu_2O system correlate with the absorption spectra and i_p spectral distribution curves of $PbN_6(Ab)$ and Cu_2O [8, 16],

(3) the formation of U_p of the $PbN_6(Ab)$–Cu_2O systems in the spectral regions of light absorption by $PbN_6(Ab)$ and Cu_2O is evidence of the separation of nonequilibrium charge carriers on the contact under irradiation [2, 4–10].

Table 2. Rate constants for photolysis of $PbN_6(Ab)$ and the $PbN_6(Ab)$–Cu_2O system calculated from the kinetic curves of the rate of photolysis (k_{1p}) and diffuse reflectance spectra (k_{1dr}); I is the intensity of incident light (quanta cm^{-2} s^{-1})

$I \times 10^{-15}$	$PbN_6(Ab)$		PbN_6 (Ab) – Cu_2O	
	$k_{1p} \times 10^2$	$k_{1dr} \times 10^2$	$k_{1p} \times 10^2$	$k_{1dr} \times 10^2$
0.795	4.15 ± 0.24	4.22 ± 0.32	4.50 ± 0.14	4.24 ± 0.11
1.27	4.75 ± 0.48	4.52 ± 0.61	4.20 ± 0.09	4.80 ± 0.44
2.00	4.87 ± 0.27	4.67 ± 0.44	4.36 ± 0.13	4.63 ± 0.34
3.17	5.93 ± 0.56	5.69 ± 0.61	5.80 ± 0.50	5.30 ± 0.48
5.56	3.50 ± 0.23	3.66 ± 0.17		

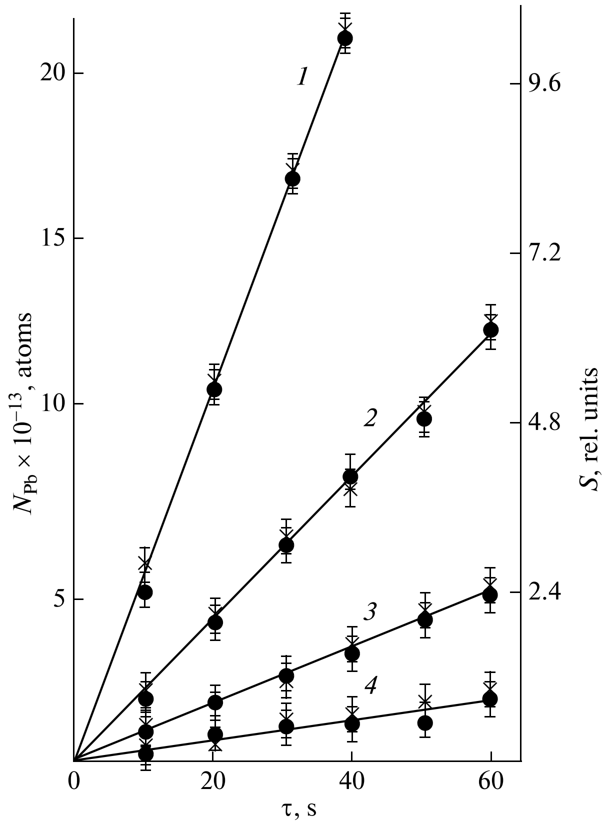

Fig. 4. Time dependences of the amounts of photolytic lead (N) and areas (S) corresponding to changes in diffuse reflectance from $PbN_6(Ab)$–Cu_2O at various incident light intensities, (*1*) 3.17×10^{15}, (*2*) 2×10^{15}, (*3*) 1.27×10^{15}, and (*4*) 7.85×10^{14} cm^{-2} s^{-1}, $\lambda = 380$ nm.

Photochemical manifestations of photoelectric processes in the $PbN_6(Ab)$–Cu_2O system can be caused by redistribution of electron-hole pairs generated by light in the region of spatial charge of the contacting partners under contact field action. As a result of this redistribution, Cu_2O admixtures can act as donors or acceptors of nonequilibrium charge carriers with respect to lead azide. According to the ratio between the thermoelectronic work functions of the contacting partners (Table 3) [8, 15, 16], rectification effects on current-voltage characteristics and equal over the whole spectrum but positive on the side of $PbN_6(Ab)$ U_p had to be expected when isolated $PbN_6(Ab)$ and Cu_2O particles approached each other.

We found that noticeable rectification effects were absent on current-voltage characteristics of the $PbN_6(Ab)$–Cu_2O system, and the sign of U_p on the side of $PbN_6(Ab)$ measured under atmospheric conditions and in a vacuum for the $PbN_6(Ab)$–Cu_2O system was negative (Fig. 1) and its value did not correspond to that expected from the ratio between the work functions of the contacting partners. This and the results of contact potential difference (Table 3) [15], capacitor photo-EMF [17], external photoemission [18], and V_p, i_p, and U_p measurements for PbN_6(Ab) and PbN_6(Ab)–Cu_2O are evidence of a considerable concentration of and important role played by intrinsic surface electronic states (T_s^-) of lead azide and contact surface electronic states (T_c^+) between lead azide and copper(I) oxide during the redistribution of charge carriers on contacts in the dark and under irradiation. When contacts between PbN_6(Ab) and Cu_2O are formed, exchange of equilibrium charge carriers occurs until thermodynamic equilibrium is established in the system. A diagram of the energy zones of the PbN_6(Ab)–Cu_2O system is given in Fig. 5.

The action of light in the region of intrinsic absorption of lead azide on the PbN_6(Ab)–Cu_2O system causes intense generation of electron-hole pairs in lead azide and the semiconductor (Fig. 5, transitions *1*, *2*),

$$N_3^- \rightarrow p + e.$$

Since the quantum yield of the photolysis of the PbN_6(Ab)–Cu_2O system is 0.002–0.010 at exposure $\tau \leq 60$ s, part of generated charge carriers recombine (Fig. 5, transitions *3*),

$$T^+ + e \rightarrow T^0 + p \rightarrow T^+,$$

where T^+ is the recombination center.

Pairs of nonequilibrium charge carriers generated in the spatial charge regions of lead azide and copper(I) oxide are redistributed in the contact field formed because of the noncoincidence of the work functions of the contacting partners (Table 3) and the presence of T_s^- and T_c^+. Nonequilibrium holes from the valence band of lead azide and nonequilibrium electrons from the conduction band of Cu_2O pass to the T_s^- and T_c^+ levels,

$$T_s^- + p \rightarrow T_s^0,$$

$$T_c^+ + e \rightarrow T_c^0.$$

Electrons and holes settled on the T_s^- and T_c^+ levels can recombine or participate in exchange with the nearest semiconductor and lead azide bands. When the PbN_6(Ab)–Cu_2O system is exposed to light in the region of absorption of Cu_2O, this causes intense generation of electron-hole pairs in the semiconductor (Fig. 5, transition *2*). Nonequilibrium charge carriers generated in the spatial charge region of Cu_2O are redistributed in contact field with the transition of electrons from the conduction band of the semiconductor to the T_c^+ levels. The sign of U_p (Fig. 1) on the side of lead azide in the PbN_6(Ab)–Cu_2O system is evidence of the possibility of the existence of the transitions under consideration. Simultaneously with these transitions, which result in the formation of U_p and shift energy levels of the contacting partners, there are fluxes of equilibrium charge carriers. As a result, the concentration of holes in the spatial charge region of lead azide (in contact with Cu_2O) changes compared with their concentration in pure azide.

Table 3. Contact potential difference (V) between PbN_6(Ab), Cu_2O, and reference platinum electrode at $T = 293$ K

Sample	1×10^5 Pa	1×10^{-5} Pa
PbN_6(Ab)	−0.34	−0.20
Cu_2O	+0.31	+0.28

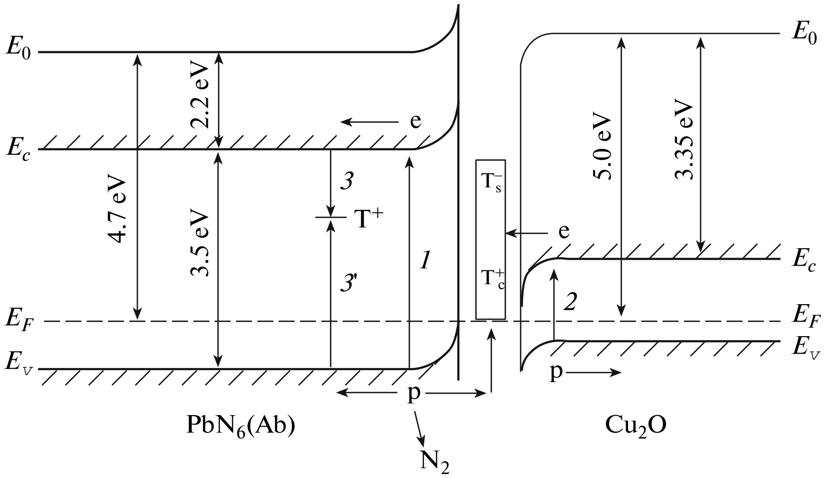

Fig. 5. Diagram of the energy bands of the PbN_6(Ab)–Cu_2O heterosystem; E_V is the top of the valence band, E_c is the bottom of the conduction band, E_F is the Fermi level, and E_0 is the level of the vacuum; T^+ is the recombination center.

The resulting change in the concentration of holes in the spatial charge region of lead azide causes the corresponding decrease in i_p and U_p in the region of intrinsic azide absorption and the appearance of i_p and photolysis in the long-wave spectrum region (Fig. 1) corresponding to the region of absorption and photoelectric sensitivity of Cu_2O according to the reactions of nitrogen formation assumed for heavy metal azides [19],

$$p + V_c^- \rightarrow V_c^0 + p \rightarrow V_c^+ \rightarrow 3N_2 + 2V_a^+ + V_c^-,$$

where V_c^- and V_a^+ and the cationic and anionic vacancies. In the photolysis of the PbN_6(Ab)–Cu_2O system, photolytic lead is formed simultaneously with the release of nitrogen. We assume that T_s^- and T_c^+ are centers of the formation of photolytic metal with the participation of mobile anionic vacancies (lead azide is disordered according to Schottky [20]),

$$T_c^0 + 2V_a^+ \rightarrow (T_c \cdot 2V_a)^{2+} + 2e \rightarrow \ldots \rightarrow (T_cPb_n)^0,$$

$$T_s^0 + 2V_a^+ \rightarrow (T_s \cdot 2V_a)^{2+} + 2e \rightarrow \ldots \rightarrow (T_sPb_n)^0.$$

The observed decrease in V_p in the initial region of the kinetic curve during and after preliminary exposure of samples (Fig. 2) substantiates irreversible consumption of surface centers. During the growth of photolytic metal particles, lead azide–lead (photolysis product) microheterogeneous systems are formed [8–10]. When light from the region of intrinsic absorption of lead azide acts on the PbN_6(Ab)–Pb systems, pairs of carriers generated in the spatial charge region of lead azide are redistributed in the contact field formed because of the noncoincidence between the thermoelectronic work functions of lead azide and photolytic lead with the transition of nonequilibrium electrons from the conduction band of PbN_6(Ab) into lead. Simultaneously, there occurs the photoemission of holes from lead to the valence band of lead azide. These processes can stimulate the diffusion of anionic vacancies and electrons to growing particles and, as a consequence, increase their size,

$$(T_cPb_n)^0 + 2V_a^+ \rightarrow (T_cPb_n \cdot 2V_a)^{2+} + 2e \rightarrow (T_cPb_{n+1})^0,$$

$$(T_sPb_n)^0 + 2V_a^+ \rightarrow (T_sPb_n \cdot 2V_a)^{2+} + 2e \rightarrow (T_sPb_{n+1})^0.$$

As a result, the concentration of holes in the spatial charge region of lead azide and V_p of the PbN_6(Ab)–Cu_2O system increase (Fig. 1, region III). During photolysis, the separation boundary of PbN_6(Ab)–Cu_2O contacts becomes covered by a photolytic lead layer, and, at large degrees of transformation, photochemical processes in the PbN_6(Ab)–Cu_2O system are to a substantial extent determined by photoelectric processes at the lead azide–lead (photolysis product)–copper(I) oxide boundary.

To determine the limiting stage of the growth of photolytic lead particles, we estimated time during which a mobile anionic vacancy neutralizes an electron or diffuses to a neutral center. The relaxation time of the drift mechanism is the Maxwell relaxation time [21],

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

where ε is the permittivity ($\varepsilon_{PbN_6} = 6$) and σ is the specific conductivity at $T = 293$ K ($\sigma_{PbN_6} \approx 1 \times 10^{-12}\ \Omega^{-1}\ cm^{-1}$). This gives $\tau_i = 0.4$ s. The rate constant for photolysis is then $k^I = 2.5\ s^{-1}$.

The relaxation time of a diffusion process can be estimated as [21]

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

where e is the charge of the electron, a is the lattice constant ($a_{PbN_6} = 8 \times 10^{-10}$ cm), $T = 293$ K, and k_B is the Boltzmann constant. At $T = 293$ K, $\tau_d = 80$ s. The rate constant of photolysis is then $k^{II} \approx 1.25 \times 10^{-2}\ s^{-1}$. Satisfactory coincidence of the rate constants for photolysis (Table 2) and k^{II} leads us to suggest that the limiting stage of the photolysis of PbN_6(Ab)–Cu_2O is the diffusion of anionic vacancies to a neutral center.

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