

Radiation Effects and Interphase Interactions in Ohmic and Barrier Contacts to Indium Phosphide as Induced by Rapid Thermal Annealing and Irradiation with γ -Ray ^{60}Co Photons

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Abstract—The radiation resistance of Au–Pd–Ti–Pd– n^{++} –InP ohmic contacts and Au–TiB_x– n – n^+ – n^{++} –InP barrier contacts—both initial and subjected to a rapid thermal annealing and irradiated with ^{60}Co γ -ray photons with doses as high as 10^9 R—has been studied. Before and after external effects, the electrical characteristics of the barrier and ohmic contacts, distribution profiles for components, and phase composition in the metallization layers have been measured. In ohmic Pd–Ti–Pd–Au contacts subjected to rapid thermal annealing and irradiation, a significant distortion of the layered structure of metallization occurs; this distortion is caused by the thermal and irradiation-stimulated transport of Pd over the grain boundaries in polycrystalline Ti and Au films. However, the specific contact resistance ρ_c does not change appreciably, which is related to a comparatively unvaried composition of the contact-forming layer at the Pd– n^+ –InP interface. In the initial sample and the sample subjected to the rapid thermal annealing at $T = 400^\circ\text{C}$ with the Au–TiB_x– n – n^+ – n^{++} –InP barrier contacts and irradiated with the dose as high as 2×10^8 R, a layered structure of metallization is retained. After irradiation with the dose as high as 10^9 R, in the samples subjected to a rapid thermal annealing at $T = 400^\circ\text{C}$, the layered structure of metallization becomes completely distorted; however, this structure is retained in the initial sample. The electrical properties of the contact structure appreciably degrade only after irradiation of the sample preliminarily subjected to a rapid thermal annealing at $T = 400^\circ\text{C}$.

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1. INTRODUCTION

Indium phosphide is one of a few semiconductor materials used for fabrication of Gunn diodes for millimeter and submillimeter wavelength regions [1–6]. Interest in the component base of ultrahigh-frequency (microwave) electronics in the millimeter wavelength range on the basis of InP, being one of the most preferable materials for fabrication of Gunn diodes, is related to low losses of microwave power in the case of transmission of signals under unfavorable conditions accompanied with increasing the volume and rate of transfer of information, as well as with a number of advantages caused by the electrical properties of InP. Featuring a higher drift mobility of electrons and a higher heat conductivity than does GaAs, InP is promising for fabrication of high-efficiency Gunn diodes generating in the short millimeter-wavelength region. However, realization of these advantages of indium phosphide significantly depends on the quality

of ohmic contacts and the quality of the barrier contacts in the case of microwave diodes with the Schottky barrier and field-effect transistors. The thermal stability of the contact metallization to InP has been adequately studied, whereas the effect of ionizing radiation on the properties of ohmic and barrier contacts has been poorly investigated. It is also noteworthy that it has now been established that the parameters of ohmic and barrier contacts to InP depend on the contacting metals, treatment of the surface, temperature conditions of formation of contacts (these conditions govern the processes of interphase diffusion (mass transport)), and chemical reactions, on which, in turn, the phase composition of contacting materials and the properties of transition layers at the metal–InP interfaces depend.

In order to increase the thermal stability of contacts in the layers of metallization between the capping layer (most often Au) and the contact-forming layer,

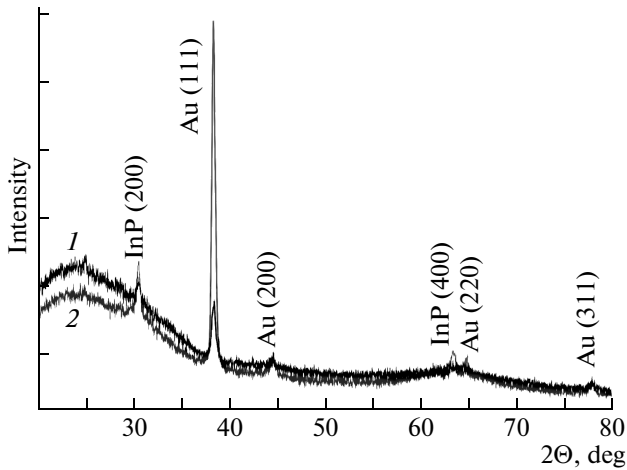


Fig. 1. The X-ray diffraction pattern for the Au–TiB_x–InP contact structures: (1) an initial sample irradiated with ⁶⁰Co γ-ray photons at a dose of 2×10^8 R and (2) a sample subjected to RTA at 400°C and irradiated with a dose of 2×10^8 R.

buffer layers (diffusion barriers) are introduced. As a rule, all the films of metals (alloys) incorporated into the contact metallization are polycrystalline. Therefore, the long-time operation of such contacts in InP-based devices at an elevated temperature and at a high dissipated power is found to be limited by intergrain diffusion in the metallization components and by mass transport at the metal–semiconductor interface.

The thermal stability of contacts can be improved if the diffusion (mass transport of the components of metallization and semiconductor) over the grain boundaries is excluded or appreciably retarded. To this end, diffusion barriers composed of amorphous (quasi-amorphous) interstitial alloys were suggested for contacts to wide-gap semiconductors [7, 8]. Nitrides, borides, and tungstenides of refractory metals are such alloys. It was previously shown [8, 9] that barrier Au–TiB_x–*n*–*n*⁺–*n*⁺⁺–InP contacts are thermally stable at temperatures as high as 600°C. An increase in the mass transport after annealing at $T = 800^\circ\text{C}$ is related to relaxation of internal stresses in the contact metallization due to cracking of the latter. At the same time, the layered metallization structure and barrier properties are retained in the cracked fragments of the sample. It is noteworthy that, after thermal annealing at temperatures as high as $T = 800^\circ\text{C}$, the TiB_x film remains quasi-amorphous, which ensures high thermal stability of contacts. However, radiation resistance of contacts to InP with an amorphous (quasi-amorphous) TiB_x barrier layer and ohmic Au–Pd–Ti–Pd–*n*⁺⁺–InP contacts formed by polycrystalline metallization layers has not been studied.

The aim of this study consisted in investigation of radiation resistance of the Au–TiB_x–*n*–*n*⁺–*n*⁺⁺–InP barrier contacts and Au–Pd–Ti–Pd–*n*–InP ohmic

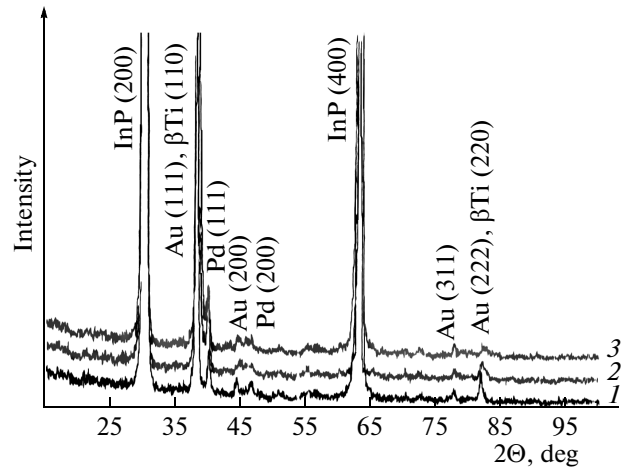


Fig. 2. The X-ray diffraction patterns for (1) an initial Au–Pd–Ti–Pd–InP contact structure and the structure subjected to RTA at (2) 300°C and (3) 400°C.

contacts irradiated with ⁶⁰Co γ-ray photons with doses as high as 10^9 R in original contact structures and structures subjected to rapid thermal annealing (RTA) of contact structures.

2. EXPERIMENTAL

The Au–TiB_x–*n*–*n*⁺–*n*⁺⁺–InP barrier structure was formed by sputtering of TiB_x (50 nm) and Au (50 nm) in one technological operation. Ohmic contacts were formed by sequential resistive sputtering deposition of Pd (30 nm), Ti (50 nm), Pd (100 nm), and Au (100 nm) in vacuum with pressure of $\sim 10^{-7}$ Torr. In both cases, metallization was formed on the surface of the substrate heated to 300°C and the surface preliminarily subjected to photonic cleaning in the case of *n*⁺⁺–InP (ohmic contacts) or *n*–*n*⁺–InP layer (barrier contacts). The concentration of the doping impurity in *n*⁺⁺–InP was $\sim 10^{18}$ cm⁻³, the substrate thickness was ~ 350 μm, and the impurity concentration in the active epitaxial layers with the thickness $\sim (2-3)$ μm was no higher than 8×10^{16} cm⁻³ and no higher than 5×10^{17} cm⁻³ in the buffer *n*⁺–InP layers. TiB_x films with the size of ordered regions in the film on the order or smaller than 3 nm were obtained at a discharge current of 0.4 A and made it possible to form quasi-amorphous TiB_x films; the structure of these films did not change as a result of RTA at $T = 300^\circ\text{C}$ (400°C) and after irradiation with γ-ray photons with a dose as high as 2×10^8 R, which is confirmed by the X-ray diffraction patterns (Fig. 1) of the Au–TiB_x–*n*–*n*⁺–*n*⁺⁺–InP contact structures in the original sample and the sample subjected to an RTA at 400°C.

The phase composition of ohmic metallization after all external effects practically remains unchanged. The Pd, Ti, and Au films remained polycrystalline, which is confirmed by the typical X-ray diffraction patterns (see Fig. 2) for the initial samples

Table 1. The influence of rapid thermal annealing and irradiation with the ^{60}Co γ -ray photons on the specific resistance of ohmic contacts to n^{++} -InP

Contacts	$\rho_c, \Omega \text{ cm}^2$		
	initial	after RTA 300°C	after RTA 400°C
Au-Pd-Ti-Pd (initial)	$2 \times 10^{-3} - 5 \times 10^{-3}$	$2 \times 10^{-3} - 5 \times 10^{-3}$	$5 \times 10^{-3} - 7 \times 10^{-3}$
Au-Pd-Ti-Pd (after irradiation, 10^8 R)	$4 \times 10^{-3} - 5 \times 10^{-3}$	$2 \times 10^{-3} - 3 \times 10^{-3}$	$5 \times 10^{-3} - 8 \times 10^{-3}$

and the samples subjected to RTA at 300°C (400°C). Irradiation of the original sample (and also the sample subjected to an RTA at 400°C) with a dose as high as 10^8 R did not change the shape of the diffraction pattern.

We studied two types of samples: diode structures and test structures with the area $1 \times 1 \text{ cm}^2$ for measurements of the distribution profiles for components in the metallization layers by the method of electron Auger spectroscopy and X-ray phase analysis. In order to measure the specific contact resistance ρ_c , we used the transmission line method (TLM) [10]. Diode structures with a diameter of 100 μm were formed using photolithography. These structures were used in measurements of current–voltage (I – V) characteristics; the barrier height ϕ_b and the ideality factor n were calculated on the basis of these characteristics.

Some of the diode and test structures were subjected to RTA at 300°C (400°C) with subsequent irradiation with ^{60}Co γ -ray photons; the doses were 10^{18} , 2×10^8 , and 10^9 R . The initial (not subjected to RTA) samples were also irradiated with these doses.

Before and after external effects, we measured the electrical characteristics of the barrier and ohmic contacts, distribution profiles for components, phase composition in the metallization layers, and surface morphology for the top metallization layer (Au).

3. RESULTS AND DISCUSSION

In Fig. 3, we show the results of layer-by-layer Auger profiling of the Au–Pd–Ti–Pd– n^{++} -InP before and after RTA and irradiation with the ^{60}Co γ -ray photons at doses as high as 10^8 R . The experimental data indicate that there is a layered metallization structure in the initial sample and as large as 10% of oxygen in the Ti layer. An RTA at 300°C (400°C) does not appreciably change the oxygen content in the Ti film but brings about (as the RTA temperature is increased) a considerable mass transport of Pd through the polycrystalline Au film and escape of Pd to the surface of the Au film; the amount of escaped Pd is 12% after RTA at 300°C and 26% after RTA at 400°C. In addition, the extent of the transition layer at the Pd– n^{++} -InP interface and smearedness of this layer on the InP side increase after RTA at 400°C, which indicates that interaction between Pd and InP is intensified.

Irradiation with ^{60}Co γ -ray photons was conducive to enhancement of mass transport of Pd into indium phosphide and to its surface through the Au film. In this case, as much as 10% of Pd is observed on the surface of the Au film with an insignificant content of oxygen. As can be seen from Fig. 3, the mass transport of Pd is enhanced in the samples subjected to RTA at 300°C (400°C) and irradiated with a dose as high as 10^8 R . In this case, however, the content of oxygen in the Ti film remains practically unchanged. Appearance of new phases was not observed in the X-ray diffraction patterns of all irradiated samples.

It is essential that, in the contact structures subjected to RTA and irradiation, we observe a significant violation of the layered structure of metallization, which can be related to the thermal- and radiation-stimulated mass transport of Pd, which is enhanced by possible relaxation of internal stresses; this relaxation was previously observed in the III–V multilayered structures [11].

The effect of RTA and irradiation with ^{60}Co γ -ray photons on the value of ρ_c is represented in Table 1.

It can be seen from the data listed in Table 1 that the ohmic contact to n^{++} -InP is formed in the course of deposition of metals onto the InP substrate heated to 300°C. The subsequent RTA at 300°C (400°C) practically did not affect the value of ρ_c , which is in good agreement with the data of the X-ray phase analysis and Auger profiling; these data indicate that there are relatively slight changes in the region of the contact-forming layer. A slight increase in ρ_c after RTA at 400°C can be caused by an increase in the concentration of oxygen and its spatial nonuniform distribution in the contact-forming layer and also by the more nonuniform (compared to the initial samples and the samples subjected to RTA at 300°C) distribution of the P and In atoms in the transition layer.

As can be seen from Table 1, irradiation did not appreciably change the values of ρ_c in either of samples, reduced slightly the spread of ρ_c in the initial samples and the samples subjected to RTA at $T = 300^\circ\text{C}$, and led to a slight increase in ρ_c in the samples subjected to RTA at 400°C, apparently due to the radiation-stimulated relaxation of internal stresses in these samples.

The data of Auger profiling for Au–TiB_x– n - n^+ – n^{++} -InP contacts are shown in Fig. 4; it can be seen that, both in the initial sample and in the sample sub-

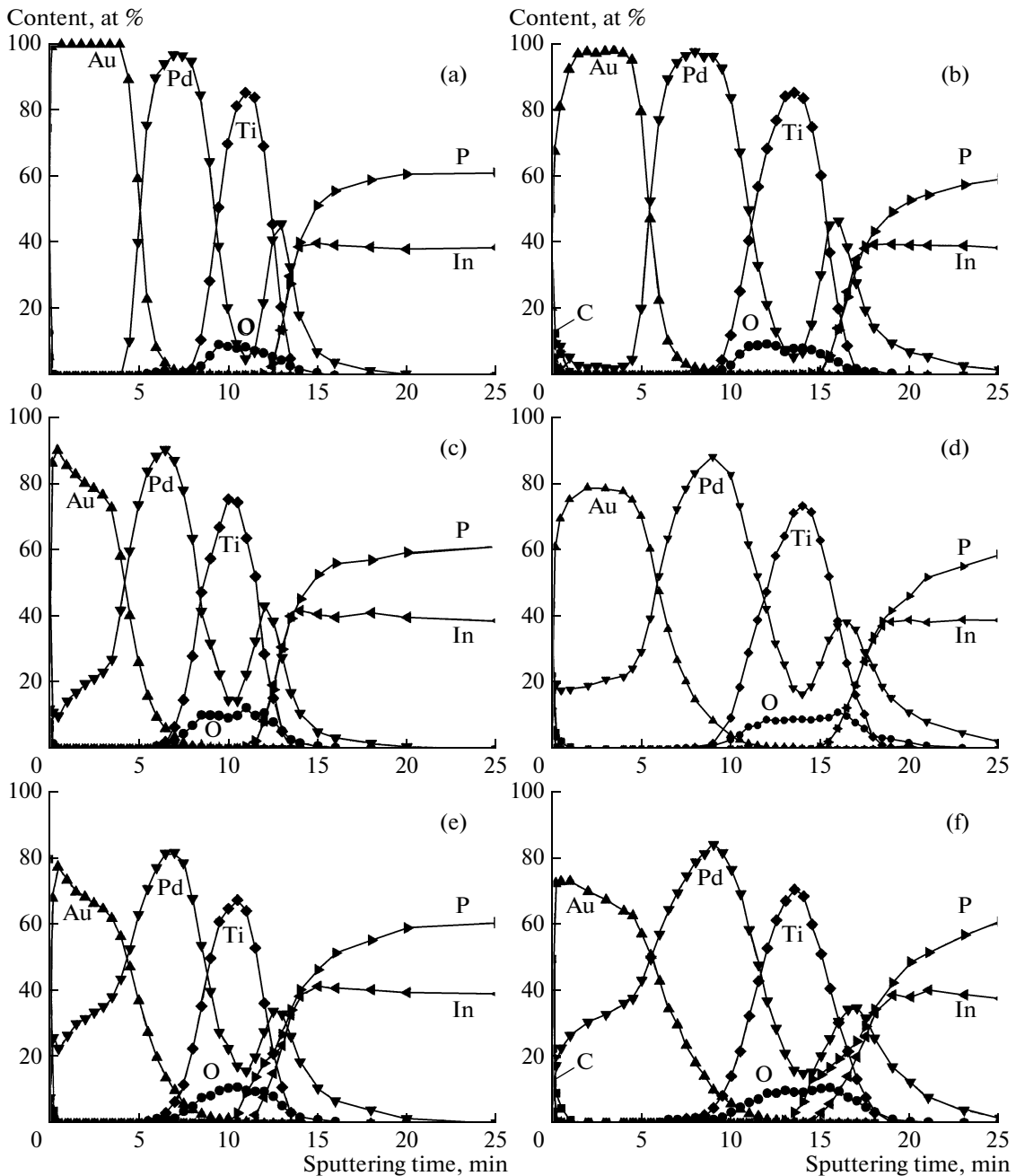


Fig. 3. Distribution profiles for components in the Au–Pd–Ti–Pd– n^{++} –InP contact before and after RTA at $T = 300$ and 400°C (a, c, e) and for the same samples irradiated with ^{60}Co γ -ray photons at a dose of 10^8 R (b, d, f).

jected to RTA at $T = 400^\circ\text{C}$ and then irradiated with the dose 2×10^8 R, a layered structure of metallization is clearly observed. This structure is disrupted owing to an appreciable increase in the oxygen concentration as a result of irradiation of both types of samples with a dose of 10^9 R. It is noteworthy that, in the sample subjected to an RTA and irradiated with the dose of 10^9 R, the layered structure of metallization is completely disrupted, while the morphology of the surface of the Au film becomes microporous (Fig. 5c), rather than

fine-grained as in the sample subjected to RTA; unirradiated; and irradiated with the dose of 2×10^8 R (Figs. 5a, 5b).

It is worth noting that the data on the distribution profiles for the components in the metallization layers in initial samples subjected to RTA and irradiated with a dose of 2×10^8 R correlate with the results of X-ray phase analysis; it follows also from the latter that the quasi-amorphous phase of TiB_x is retained in the samples under analysis. In addition, judging from the data

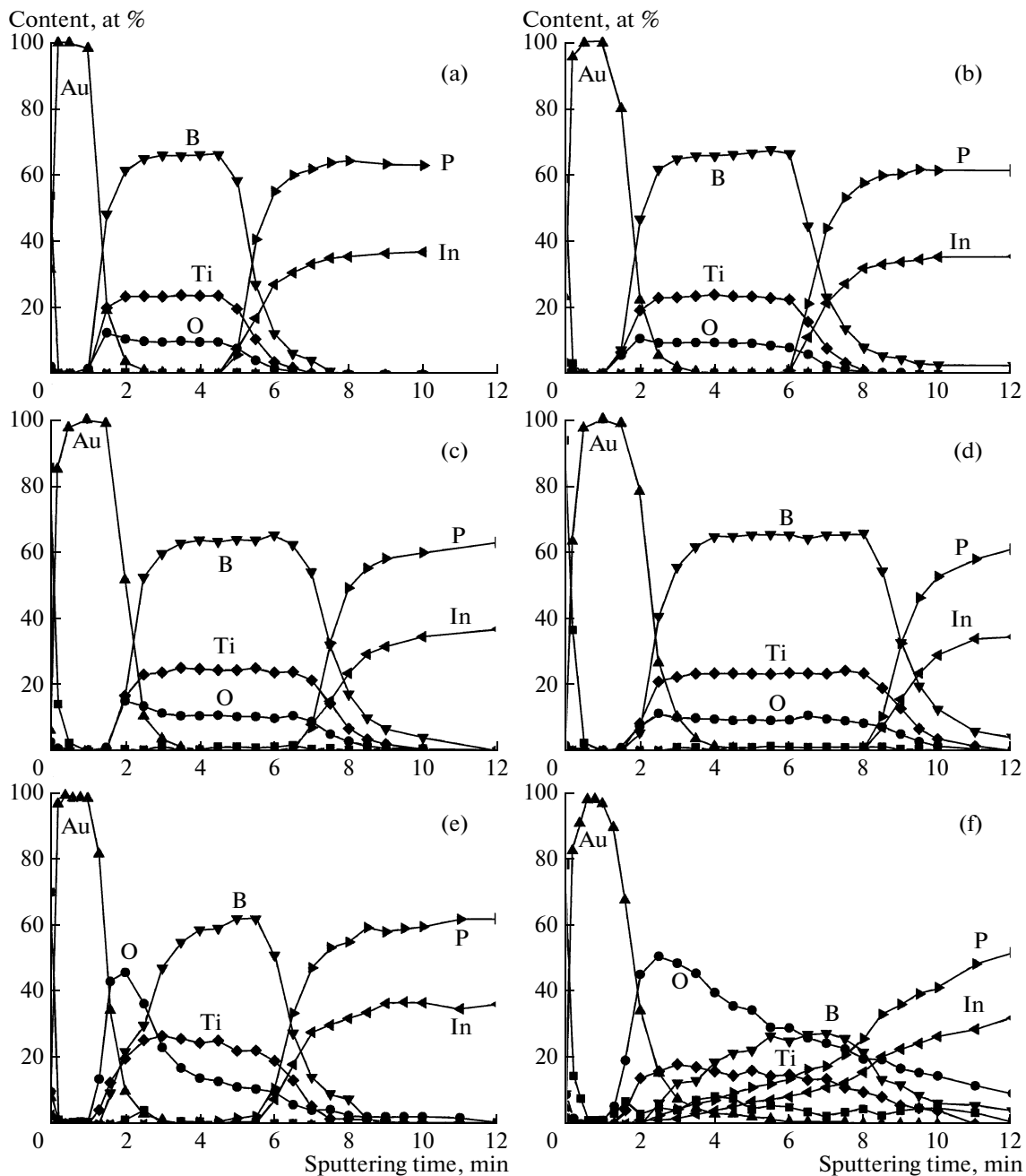


Fig. 4. The distribution profile for components in the Au-TiB_x-n-n⁺-n⁺⁺-InP before and after RTA at $T = 400^\circ\text{C}$ (a, b), before and after RTA at $T = 400^\circ\text{C}$ and after irradiation with ^{60}Co γ -ray photons at doses 2×10^8 R (c, d), and before and after RTA at $T = 400^\circ\text{C}$ and after irradiation with ^{60}Co γ -ray photons at the dose of 10^9 R (e, f).

of Auger profiling, the presence of oxyborides stable to irradiation with a dose as high as 2×10^8 R cannot be excluded in a TiB_x film, which contains oxygen with a content as high as 10%. As we previously reported [12], oxidation processes in the samples of both types (initial and subjected to an RTA) irradiated with a dose as high as 10^9 R can be related to introduction of oxygen or small amount of H₂O into the samples from sur-

rounding medium. In this case, the oxidation rate increases and, since metallization layers are apparently saturated with a higher concentration of structural defects in annealed samples, the permeability of these layers to oxygen is found to be higher than for denser TiB_x layers in the initial samples irradiated with a dose of 10^9 R. It was also found that the X-ray diffraction pattern of an initial sample irradiated with

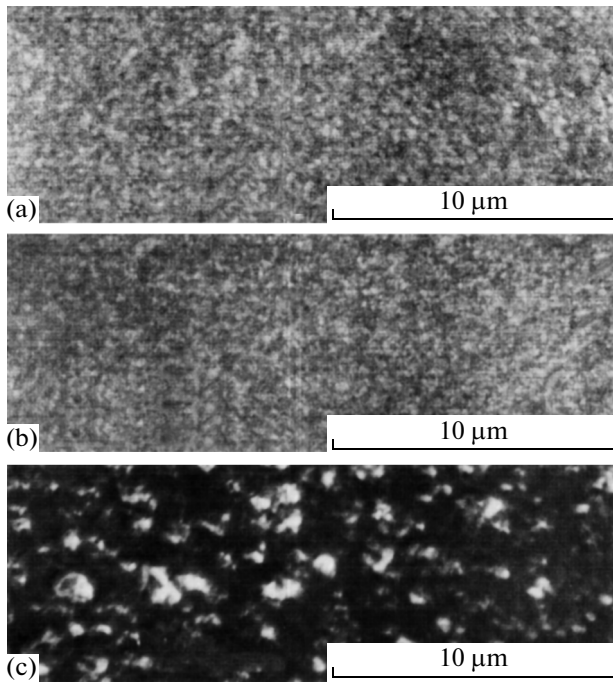


Fig. 5. Morphology of the surface of (a) an initial Au—TiB_x—InP test structure, (b) this structure after RTA at $T = 400^\circ\text{C}$, and (c) the structure subjected to RTA at $T = 400^\circ\text{C}$ and irradiated with the dose 10^9 R.

10^9 R and of the sample preliminarily subjected to an RTA at 400°C and then irradiated with a dose of 10^9 R differ from each other (Fig. 6). In this case, in the X-ray diffraction pattern of the latter sample, a reflection corresponding to the AuIn₂ phase is observed; this may indicate that the TiB_x diffusion barrier is disrupted owing to irradiation. In contrast, new phases are not observed in the X-ray diffraction pattern for the irradiated initial sample. It is noteworthy that a certain smearedness of the reflection for the AuIn₂ phase indicates that this phase includes not only AuIn₂.

Table 2. The influence of rapid thermal annealing and irradiation with ^{60}Co γ -ray photons on φ_B and n for the Au—TiB_x— n - n^+ — n^{++} —InP

Contacts	φ_B , V	n
Au—TiB _x (initial)	0.49–0.5	1.1–1.15
Au—TiB _x (RTA 400°C)	0.49–0.51	1.2–1.25
Au—TiB _x (initial, irradiation 10^9 R)	0.5–0.51	1.1–1.15
Au—TiB _x (RTA 400°C , irradiation 2×10^8 R)	0.5–0.52	1.15–1.22
Au—TiB _x (initial, irradiation 2×10^8 R)	0.49–0.54	1.25–1.5
Au—TiB _x (RTA 400°C , irradiation 10^9 R)	Linear I – V characteristic	

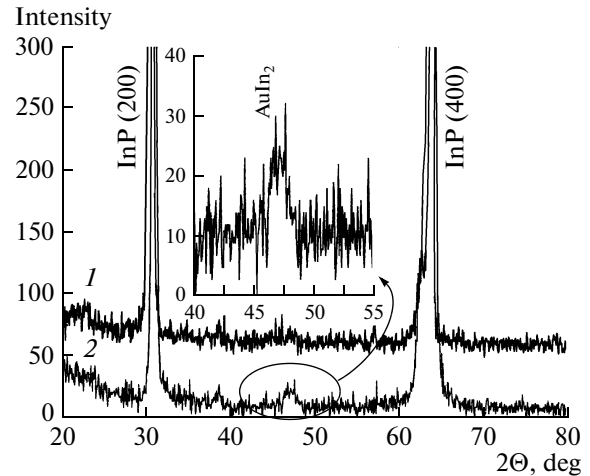


Fig. 6. The X-ray diffraction patterns for the following Au—TiB_x—InP contact structures: (1) the initial structure irradiated with the ^{60}Co γ -ray photons at the dose of 10^9 R and (2) the structure subjected to RTA at $T = 400^\circ\text{C}$ and then irradiated with the dose of 10^9 R. In the inset, we show a portion ($\times 7.5$) of the diffraction pattern with reflection from the AuIn₂ phase.

The height of the Schottky barrier φ_b and the ideality factor n for the structures under study are listed in Table 2.

It can be seen from the data listed in Table 2 that radical changes in the parameters are observed only for the sample subjected to an RTA and irradiated with a dose of 10^9 R. In this case, the shape of the I – V characteristic transforms from exponential to linear, which well correlates with the distribution profile for the components in the contact metallization and also with the results of the X-ray phase analysis and the data on the surface morphology of the Au film.

Comparison of processes of interphase interactions in the contact metallization composed of polycrystalline films of contact-forming and buffer metals in quasi-amorphous barrier-forming layers shows that polycrystalline metal films are found to be diffusion-penetrable both as a result of RTA and after irradiation with high doses of [gamma]-ray photons. It is noteworthy that the mass transport is more intense in irradiated samples previously subjected to an RTA than in irradiated initial structures, which is clearly seen by the example of mass transport of Pd through the Au film (Fig. 3) after each effect, except for the initial sample. The observed process is an expected confirmation of the grain-boundary diffusion in polycrystalline films of contact metallization since such films, according to available publications [13, 14], exhibit a high density of structural defects, including grain boundaries, pores, and other defects of the structure. In addition, transition regions between individual metallization layers may not only contain above-men-

tioned defects, but may also be chemically nonuniform, which also promotes the mass-transport processes enhanced by introduction of additional defects as a result of RTA and irradiation.

Interphase interactions occur differently in the Au–TiB_x–n–n⁺–n⁺⁺–InP contacts. Since TiB_x changes its phase state and does not interact with Au and InP (is chemically inert with respect to Au and InP) in the course of an RTA at 400°C and after irradiation of annealed samples with the dose of 2 × 10⁸ R (Fig. 1), the contact metallization remains thermally stable and radiation-resistant, while the barrier retains its working capacity.

The structure–phase state of metallization, the Au/TiB_x interfaces (for the initial contact) and metallization, and the Au/TiB_x and TiB_x/InP interfaces (for the sample subjected to an RTA at 400°C) change after irradiation with the dose of 10⁹ R. Since the contact metallization is a thermodynamically nonequilibrium system, introduction of point defects into it as a result of irradiation and intensification of this process in the course of irradiation of the samples preliminarily subjected to an RTA stimulates the transformation of such system into a higher equilibrium state due to activation of point defects and their interaction with dislocations, grain boundaries, pores, and other structural imperfections inherent in metal films. It was previously shown [15, 16] that, as the dose of irradiation is increased, this process can bring about pore-formation in polycrystalline films, which, in our case, are the Au films deposited onto quasi-amorphous TiB_x films, which is accompanied by intense penetration of oxygen into metal coating layers and accumulation of oxygen in both cases at the Au–TiB_x interface. However, in the sample subjected to an RTA before irradiation, oxygen diffused over the entire thickness of metallization and penetrated to an appreciable depth into the InP layer. In this case, the layered structure of metallization was found to be disrupted, while, in the presence of an appreciable amount of oxygen, effective redistribution of components in InP and TiB_x occurs that is caused by more intense removal of diffusion barriers and, consequently, by more intense mass transport enhanced by the presence of pores in the metallization layers.

4. CONCLUSIONS

Thus, the contact Au–Pd–Ti–Pd–n⁺⁺–InP metallization formed by polycrystalline Au, Pd, and Ti films; subjected to an RTA; and irradiated with ⁶⁰Co γ-ray photons exhibits a variation in the distribution profiles for the metallization components; this redistribution is caused by the thermal- and radiation-induced mass transport of Pd both at the interface with indium phosphide and through polycrystalline gold

film, which does not affect significantly the value of ρ_c since the ohmic contact was formed in the course of deposition of the Pd film onto the InP substrate heated to 300°C, while the phase composition of metallization after an RTA at 300°C (400°C) and irradiation with ⁶⁰Co γ-ray photons with the dose of 10⁸ R does not change.

The radiation resistance of the contact metallization and the Au–TiB_x–n–n⁺–n⁺⁺–InP contact formed by the TiB_x film on the InP substrate heated to 300°C is caused by the presence of the quasi-amorphous TiB_x film, which contains as much as 10% of oxygen. Similar samples subjected to the RTA at 400°C with subsequent irradiation with the ⁶⁰Co γ-ray photons with the dose were found to be radiation-resistant at a dose as high as 2 × 10⁸ R. A further increase in the dose of irradiation with γ-ray photons to 10⁹ R brings about a disruption of the layered metallization structure and complete degradation of the barrier properties of the contact.

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