

# Gold on surface of $A^{III}B^V$ crystals: effects of catalytic dissociation and anisotropic imbedding

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Received October 21, 2025

Revised October 27, 2025

Accepted October 27, 2025

Ability of metallic gold to interact chemically with semiconductor crystals  $A^{III}B^V$ , arsenides and phosphides at temperatures that do not yet allow their spontaneous dissociation is experimentally confirmed. High selectivity of the interaction relatively to crystallographic orientation of the crystal surface in contact with gold is shown. The observed catalytic effect produced by gold on dissociation of the chemical bond in these crystals is associated with presence of dimerized arsenic or phosphorous atoms on their surfaces in contact with Au. A proximity of energy positions of the lone pair electrons occupied dangling bonds of dimers of  $B^V$  elements to Fermi level in gold ensures a possibility of electron tunneling from the dimers into gold and emergence of radical single electron states on the dangling bonds of dimers. Subsequent processes leading to formation of  $B_2^V$  molecules and their separation from the crystal followed by dissolution of the liberated  $A^{III}$  atoms in gold is discussed. Each of these processes requires less activation energy than direct dissociation of the crystal. The suggested interpretation of the interaction between  $A^{III}B^V$  crystal surfaces and gold provides an explanation for the observed metal penetration into volume of crystals and the resulting crystallographic configurations of formed etching pits. A preliminary analysis of the processes that ensure an emergence of areas of direct contact between crystals and gold deposited on initially oxidized surfaces is given.

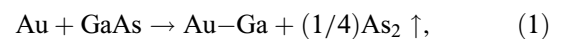
**Keywords:** semiconductors  $A^{III}B^V$ , natural oxide, surface reconstruction, dimers of  $B^V$  atoms, surface states, gold, tunneling phenomena, catalytic dissociation.

DOI: 10.61011/SC.2025.07.62473.8680

## 1. Introduction

Studies of the physical and chemical processes occurring in the area of contact between metals and semiconductor surfaces are important from a fundamental point of view and in connection with the use of such contacts (ohmic and rectifying) in semiconductor microelectronic devices. The phenomena that occur when metallic gold comes into contact with the surfaces of semiconductors  $A^{III}B^V$  are of particular interest. Due to its low resistivity, high plasticity and oxidation resistance, gold remains the most used material for creating electrical contacts in semiconductor devices based on these compounds. At the same time, it has long been observed that direct contact of gold with GaAs and InP crystals leads to their chemical destruction at temperatures that do not yet allow any manifestations of spontaneous dissociation of these compounds (up to room temperatures), and the resulting intermetallides Au–Ga(In) are embedded in crystal volume [1–5]. Contact with other metals under similar conditions does not have any pronounced effect on crystals  $A^{III}B^V$ . The observed effects have not yet been adequately explained. The need to explain them is becoming even more urgent because of the recently increasing interest in the properties of heterocompositions of nanodispersed gold with crystals  $A^{III}B^V$ , which are used in the growing of semiconductor nanowires [6–8], as well as in the creation of functional structures of nanoplasmonics [3,9,10].

Fairly detailed phenomenological descriptions of the interaction of metallic gold with GaAs surfaces of various crystallographic orientations under low-temperature heating conditions are provided in Refs. [1–3]. Formally, such an interaction can be represented by a reaction



this involves a two-stage cyclic process, the first stage of which involves the formation and separation of arsenic molecules from the surface of GaAs crystal under Au film, and the second stage involves the dissolution of exposed gallium atoms in gold. It is unusual that the dissociation of GaAs in contact with gold occurs at temperatures much lower than the temperatures of the beginning of its thermal dissociation (560–580) °C, and in conditions of complete absence of solubility of arsenic in gold. So, the apparent erosion of the (001)GaAs surface, previously purified by ion etching in vacuum before being brought into contact with gold, was detected already at 100 °C, and even at room temperature after six months [1]. Another characteristic feature of the interaction of Au with GaAs crystals is its prolonged incorporation into the crystal volume, leading to the formation of faceted etching pits filled with Au–Ga intermetallides. Thus, recent studies in Refs. [3,10] have shown that 30-minute annealing at 350 °C of an Au film on the GaAs(001) surface with a natural oxide leads to the appearance of multiple wedge-shaped etching pits

elongated along the direction  $[110]$  and formed by the polar planes  $\{111\}$  of the crystal. Such pits are filled with metal, mainly consisting of the intermetallic compound  $\text{Au}_2\text{Ga}$ .

There is no doubt that the observed decrease in the GaAs dissociation temperature is due to the catalytic action of Au in direct contact with the semiconductor. However, the mechanism of this phenomenon remains unknown. The processes occurring under conditions of contact of gold with other  $\text{A}^{\text{III}}\text{B}^{\text{V}}$  compounds have been studied to an even lesser extent. The purpose of this work is to expand the understanding of the nature of the chemical interaction of Au with the surfaces of crystals of arsenides and phosphides  $\text{A}^{\text{III}}\text{B}^{\text{V}}$  — GaAs, GaP, InAs, InP, which occurs at temperatures that do not allow spontaneous dissociation of any of these compounds. The results obtained are interpreted within the framework of our proposed model, which considers the possibility of tunneling electrons to gold from the orbitals of dangling bonds of dimerized atoms of group B<sup>V</sup> (As, P) on the crystal surface.

## 2. Experimental observations

The initial, naturally oxidized surfaces of epitaxial-quality crystal substrates were cleaned by boiling in organic solvents (chloroform, isopropyl alcohol), treated in an aqueous solution of ammonia, washed with deionized water and dried in air. Gold deposition was carried out by thermal sputtering in a vacuum chamber at a residual pressure of  $10^{-7}$  Torr. The thicknesses of the deposited Au films were 8–10 nm. Next, in the same chamber, the samples were heated to temperatures in the range (320–360) °C and kept at a set temperature from 20 to 60 min. Subsequent diagnostics of the samples was performed using a scanning electron microscope (SEM) JSM7001F (JEOL, Japan). In the case of GaAs, the studied samples were prepared on substrates with orientations (001), (111)A and (111)B. The substrates of the other studied compounds  $\text{A}^{\text{III}}\text{B}^{\text{V}}$  were oriented in the (001) plane.

### 2.1. Gold on GaAs surfaces (001), (111)A,B

An obvious evidence of the chemical interaction of gold with crystals  $\text{A}^{\text{III}}\text{B}^{\text{V}}$  is the erosion of the crystal surface with the appearance of etching pits on it, which are filled with products of the dissolution of atoms of the corresponding element  $\text{A}^{\text{III}}$  in gold. In the case of contacts Au–GaAs(001) (see Figures 1 and 2) discrete pits appear, elongated along the direction of  $[110]$  on the surface and bordered by the lateral facets (111)A and (111)B [3]. In this case, the long sides of the etching pits are formed with planes (111)B, which indicates the maximum resistance of such surfaces to the destructive action of gold. Each of the formed pits frames a wedge-shaped cluster of Au–Ga intermetallic compound, which is embedded in the crystal volume.

Considering the habitus shape of gold etching pits on GaAs(001) surfaces, it is interesting to note that pits with

a similar configuration often appear on the same surfaces when etched in solutions containing various kinds of oxidants, for example in  $\text{CH}_3\text{OH}-\text{Br}_2$  [2] or in  $\text{HCl}-\text{FeCl}_3$ . However, in this case, the etching pits, as shown in Figure 1, *c*, are always elongated along the direction  $[\bar{1}\bar{1}0]$ , and their long sides are shaped by planes (111)A, which corresponds to the minimum etching rate in solutions. electrolytes.

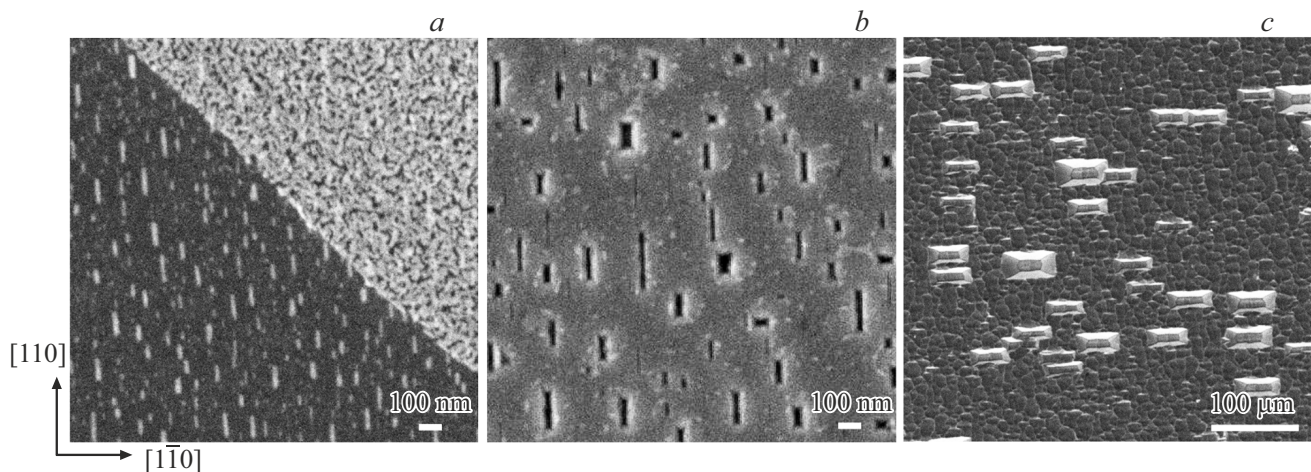
Figure 2 shows SEM images of the surfaces of GaAs crystals oriented in the planes (111)A and (111)B, after heating the 10-nanometer gold films deposited on them at 360° for 30 minutes and subsequent mechanical removal of the unreacted gold. It can be seen that on gallium surfaces (111)A clearly formed pyramidal pits with a triangular base appear, the sides of which are perpendicular to the secant planes  $\{110\}$  (Figure 2, *a*) (in this case, with a steady etching front, the inner surfaces of the pits should be decorated by facets (111)B). At the same time gold etching pits are found much less frequently on arsenic surfaces (111)B and have a smaller size and rounded shape.

### 2.2. The effect of gold on the surface of GaP(001), InAs(001) and InP(001)

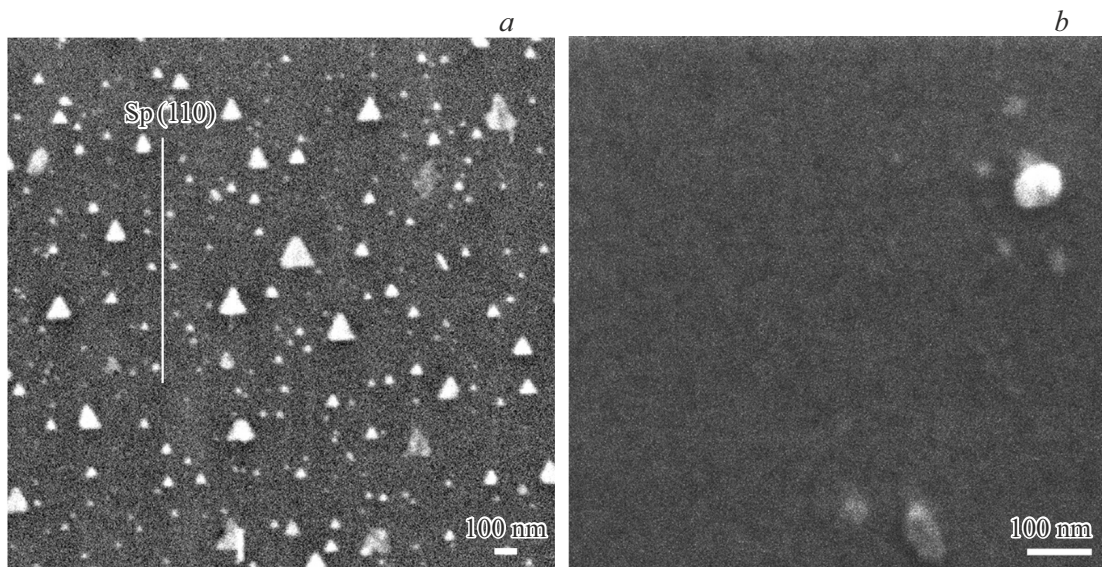
The nature of the interaction of deposited gold with the surfaces (001) of GaP crystals qualitatively coincides with the nature of its interaction with the surface GaAs(001). In both cases, during the annealing process, thin gold films disintegrate into individual clusters, in the area of contact of which with the crystal surface, discrete etching pits of a characteristic shape may appear, with a rectangular base elongated along  $[110]$ , as shown in Figure 3. The only noticeable difference is the temperature at which the etching pits begin to form and the rate at which they grow. After an hour of heating of the deposited gold at a temperature of  $\leq 350$  °C, traces of its effect on the initial surface (001)GaP become clearly distinguishable and quite numerous (see Figure 3, *b*). Under the same conditions on (001)GaAs areas of already well-formed mordants with longitudinal dimensions of pits of hundreds of nanometers are detected.

Unlike gallium crystals  $\text{A}^{\text{III}}\text{B}^{\text{V}}$ , gold films deposited on naturally oxidized InAs(001) and InP(001) surfaces do not show a pronounced tendency to disintegrate into separate clusters upon heating, remaining almost continuous and firmly bonded to the crystal after warming up. On the obtained cleavage surfaces of the formed structures, rather densely located areas of mordants with metal penetration deep into the crystals are visible, shown in Figures 4 and 5.

It is worth noting that the chemical interaction of gold with InAs and InP crystals observed here develops as a purely solid-phase process, since the minimum temperature of the eutectic transformation closest to gold in the Au–In system is 454 °C [11], which is significantly higher than the temperature of the realized process (350–360) °C. In the case of gallium compounds  $\text{A}^{\text{III}}\text{B}^{\text{V}}$ , the penetration of gold deep into the crystals can be facilitated by the



**Figure 1.** *a* — SEM image of GaAs(001) crystal surface with a 10-nanometer Au layer after annealing in vacuum for 20 min at 350 °C. The area in the left of the figure is shown after mechanical removal of gold from the surface that did not penetrate into the crystal volume; *b* — the surface of the same crystal after 30 min of annealing at 360 °C and subsequent selective removal of Au in an aqueous solution of  $K_4(Fe(CN)_6-CS(NH_2)_2)$ ; *c* — SEM image of etching pits elongated along  $[1\bar{1}0]$  on the (001)GaAs surface formed in an aqueous solution of  $FeCl_3-HCl$ . The faceting of the pits is clearly visible.

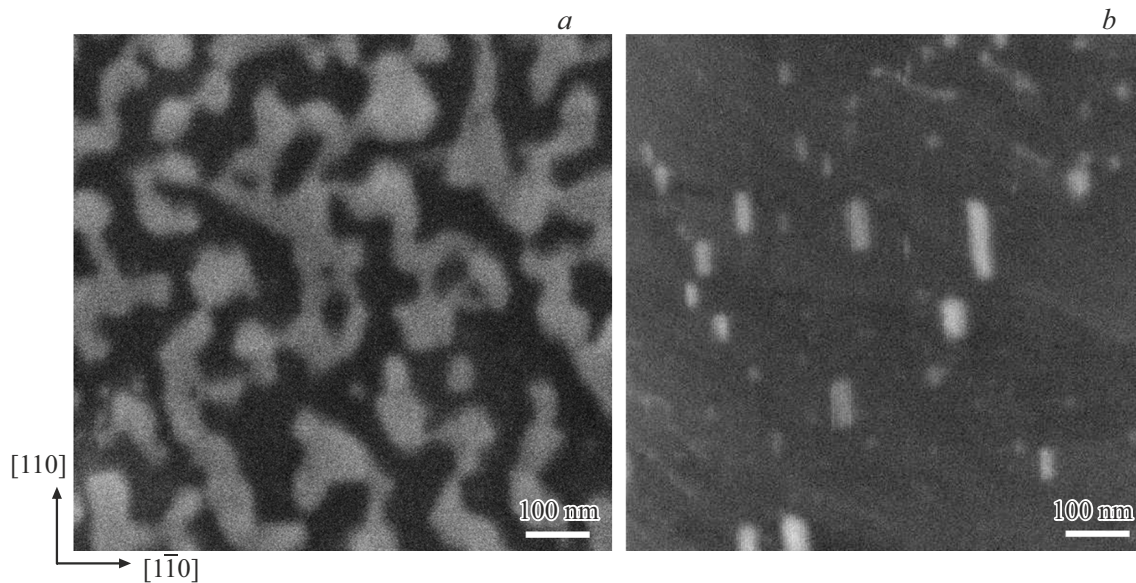


**Figure 2.** SEM images of GaAs(111)A (*a*) and GaAs(111)B (*b*) surfaces obtained after 30 min annealing of a 10 nm Au film deposited on them at 360 °C and subsequent removal of gold sections that did not react with the substrate.

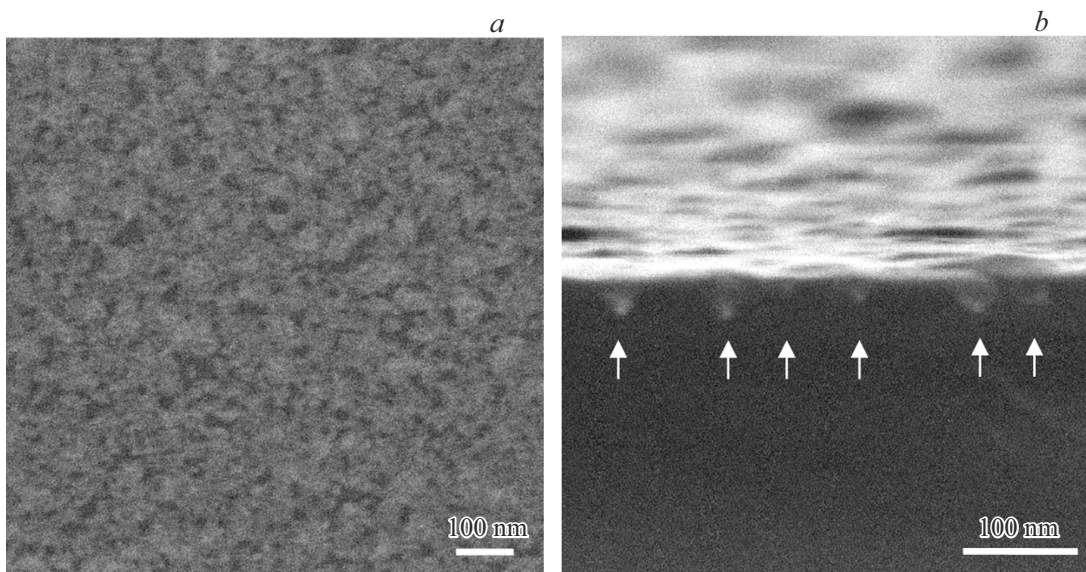
appearance of a wetting layer of the liquid phase at their interface, since the minimum eutectic temperature in the  $Au_2Ga$  system is 339 °C, and the melting point of the intermetallic compound  $Au_2Ga$  is 349 °C [12]. On the other hand, under conditions of better wettability of the oxidized surfaces of indium crystals  $A^{III}B^V$  with sprayed gold, the density of primary direct contacts with gold during heating turns out to be higher than for gallium compounds. The observed differences can be attributed to the probable presence of indium monoxide crystals  $In^V$  in the  $In_2O$  oxide layers, which is capable of disproportionation.

### 3. Discussion of experimental observations

To understand the causes of the abnormally destructive effect of gold on crystals,  $A^{III}B^V$  it is necessary to understand the atomic structure of the crystal surfaces that come into direct contact with metallic gold when heated in a vacuum, as well as the relative arrangement of the energy levels of conduction electrons in gold and electrons occupying the orbitals of broken bonds of atoms on the surfaces of semiconductor crystals in contact with gold.



**Figure 3.** SEM images obtained after an hour-long heating at 360 °C of a 10 nm thick gold layer on the GaP(001) surface: *a* — surface of the Au layer, *b* — formed underneath etching pits of the GaP crystal surface.

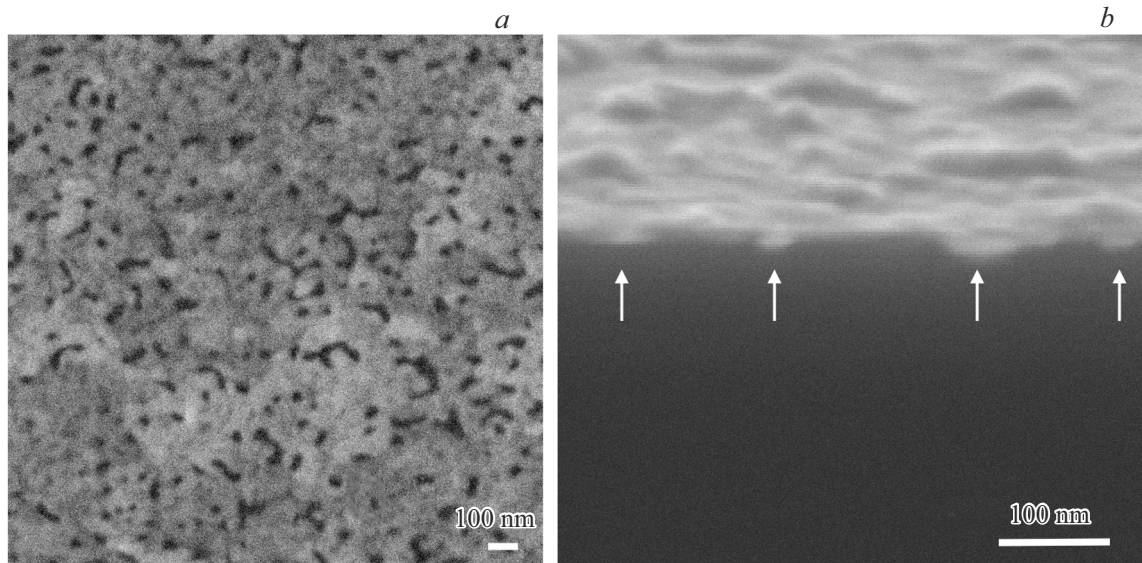


**Figure 4.** SEM images of the surface of an InAs(001) crystal with a 10-nanometer layer of gold after 20 minutes of heating in vacuum at 350 °C (*a*) and its cleavage along the plane (110), on which the areas of metal penetration are visible into the crystal volume (*b*).

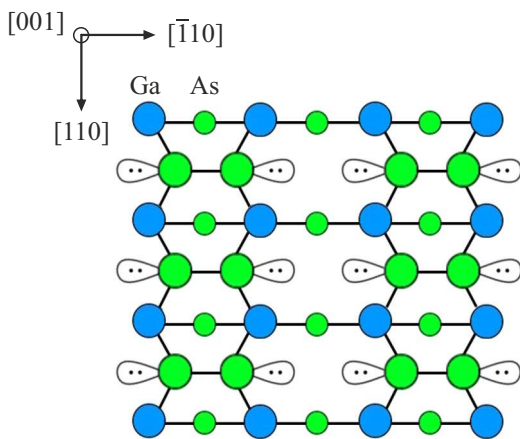
### 3.1. The state of the surfaces of crystals $A^{III}B^V$ formed when heated under a gold film

As already mentioned, Au films were deposited in vacuum on naturally oxidized crystal surfaces of  $A^{III}B^V$ . The chemical state of the oxidized surfaces GaAs(001) and InAs(001) was studied earlier in Refs. [13–15]. A thin (to the limit, monatomic) layer of elementary arsenic was found under a layer of mixed oxide of elements of groups III and V. The appearance of zero-valence arsenic can be explained by incomplete oxidation of the atoms forming the crystal lattice when the level of the chemical potential of

oxygen penetrating to the reaction front decreases as the thickness of the oxide layer increases and approaches a certain stationary value (usually  $\sim 5$  nm). When heated in vacuum to temperatures of (300–350) °C, the mixed oxide layer decomposes into solid-phase oxides of elements of group III ( $Ga_2O_3$ ,  $In_2O_3$ ,  $In_2O$ ) and arsenic oxide ( $As_4O_6$ ), which can be removed in the form of vapor at these temperatures into vacuum together with the vapor of the formed free arsenic. After the removal of both volatile components and clustering of solid-phase oxides, conditions arise for the appearance of direct local contacts of gold with a clean crystal surface, which in this case



**Figure 5.** The surface (001) of an InP crystal with a 10-nanometer gold layer after 30 min of vacuum annealing at 360 °C (a) and its cleavage along the plane (110) (it b).



**Figure 6.** Structure (2×1) of the reconstructed surface of a GaAs(001) crystal terminated by arsenic atoms forming parallel rows of dimers.

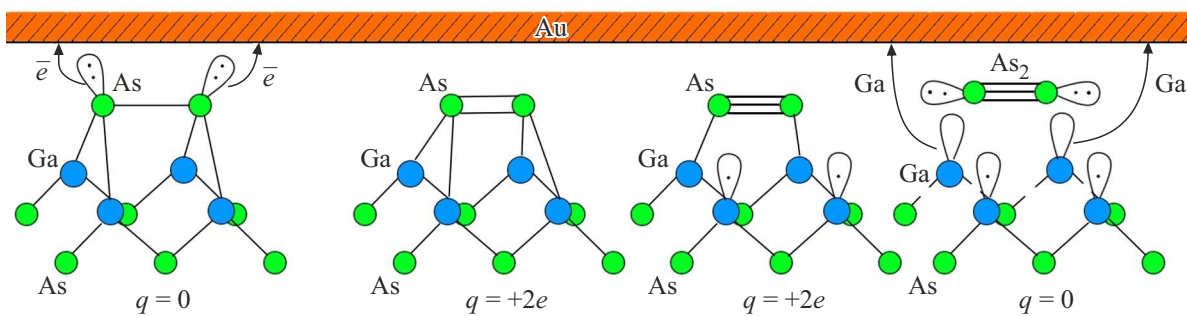
is extremely enriched in As atoms. Such surfaces must undergo reconstruction with the closure of single-electron dangling bonds between the nearest surface arsenic atoms and the formation of their dimers. For the facets (001), this corresponds to the occurrence of reconstruction (2×1) shown in Figure 6.

The arguments given here about the incomplete oxidation of the crystal components, and above all the more electronegative  $B^V$  atoms, at the final stage of oxide film formation are valid for both arsenides and phosphides  $A^{III}B^V$ , although in the latter case they do not necessarily imply the appearance of phosphorus in the elementary form at the boundary with the oxide. Nevertheless, after removing the volatile components of the oxide, the opened (001) surfaces

of the phosphide crystals should turn out to be terminated phosphorus atoms and acquire similar reconstruction.

### 3.2. Chemical bond dissociation processes in the surface layers of atoms of $A^{III}B^V$ crystals developing in contact with gold

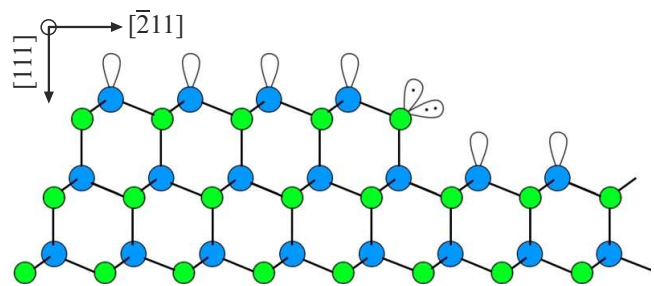
The bulk of theoretical and experimental works related to the study of band structure of the reconstructed crystal surfaces  $A^{III}B^V$  is devoted to gallium arsenide. Photoemission spectroscopy data and calculations of the positions of electron energy levels for reconstructed, As-stabilized surfaces in vacuum (001)GaAs [16–18] show that the energy levels of lone electron pairs of dimerized arsenic atoms create the most high-energy surface states lying near top of the GaAs valence band, and for some reconstruction variants (2×4) and extending into the band gap [16]. In this case, the electron energy levels corresponding to the dimer bridge bonds As-As are located deeper in the valence band of GaAs. Comparing the energy position of the top of the GaAs valence band relative to the vacuum level ( $\sim -5.49$  eV at RT and  $\sim -5.23$  eV at 360 °C [19]) with experimentally determined values of the electron work function of gold (5.1–5.47) eV [20], it is not difficult to see the proximity of the Fermi level position for conduction electrons in gold to the energy position of the lone electron pairs in arsenic dimers on (001)GaAs surface located in vacuum. In the conditions of real close contact with the metal surrounded by a vapor of free electrons (distances comparable to interatomic distances in the lattice of gold are considered), positions of the energy levels of electrons on the orbitals of the dangling bonds of atoms forming the surface of semiconductor should shift towards increasing their potential energy. This further increases the probability



**Figure 7.** Illustration of the elementary acts of the GaAs dissociation process in contact with metallic gold and the dissolution of the released gallium in it. Here  $q$  is the total positive charge of atoms bonded to the lattice within the considered grouping which is a multiple of the electron elementary charge.

of electron tunneling from lone pairs of dimerized arsenic atoms to vacant states in the conduction band of gold. In turn, such a transfer leads to the appearance of single-electron radical states of dangling bonds in paired arsenic atoms on the surface underlying gold with possibility of their subsequent closure and formation of the molecular groups  $\text{As}=\text{As}$  during the final dehybridization of valence electrons of the participating atoms. In fact, in this case, the arsenic dimer formed by atoms entering the crystal lattice passes into the state of an excited arsenic molecule, which is at the stage of desorption from its surface (see Figure 7). After the separation of the formed diatomic arsenic molecules from the crystal surface, the exposed Ga atoms, which received an uncompensated positive charge, realize the possibility of a direct transition into the volume of metallic gold with the restoration of the electron neutrality of the interface. The dissolution of gallium atoms that have come to the gold surface is followed by relaxation processes that begin another cycle of the formation of arsenic dimers and their transformation into diatomic molecules.

The proposed model of the mechanism of catalysis of gallium arsenide dissociation by metallic gold explains well the pronounced dependence of the observed processes of gold incorporation into it on the crystallographic orientation of the crystal surface (see Figures 1 and 2) and may also explain the crystallographic configuration of the etching pits that occur in this case. Indeed, comparing the results of annealing thin gold films deposited on GaAs substrates with orientations (001), (111)A and (111)B, it is clear that the introduction of gold into the crystal volume occurs most actively on substrates with orientation (001), the free surfaces of which have the highest possible density of arsenic dimers. The densities of local mordants turn out to be noticeably lower on surfaces (111)A, and especially on (111)B and, in the latter case, they devoid of cut elements. It is important to remember that the closed-packed surfaces  $\{111\}$  of  $\text{A}^{\text{III}}\text{B}^{\text{V}}$  crystals are formed by atoms having 3 bonds with the underlying layer of lattice atoms. Therefore, the etching of such surfaces almost always occurs through a layer-by-layer mechanism with the gradual separation of atoms less strongly bound to the lattice at the

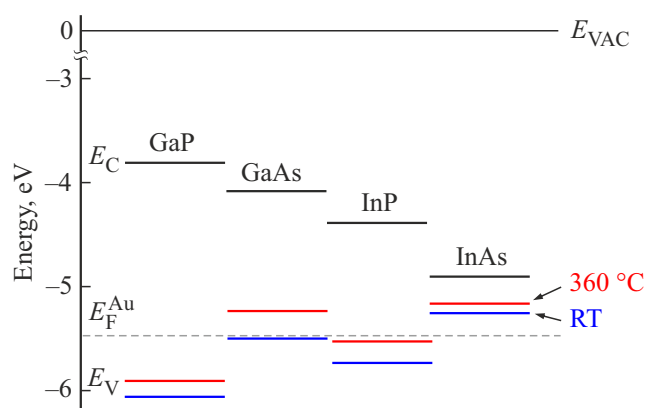


**Figure 8.** Configuration and electron population of broken bonds of atoms near the monomolecular step on the surface (111)A of the  $\text{A}^{\text{III}}\text{B}^{\text{V}}$  crystal with a sphalerite structure. View in the direction  $[011]$ .

ends of elementary steps. The configuration of the dangling bonds of such atoms at the ends of the steps extending in the directions of type  $[211]$  corresponds to their configuration on the surface (001), while the end of the step is formed by atoms of the opposite type to the atoms emerging on the etched surface  $\{111\}$  (Figure 8).

Therefore, a layer-by-layer gold etching can develop on non-ideal facets (111)A having elementary steps and areas of their generation (dislocation yields) according to the catalytic mechanism of GaAs dissociation with the participation of arsenic dimers considered here, whereas there are no conditions for such etching on surfaces (111)B. This also explains the characteristic shape of local mordants produced by gold etchings observed on the (001) surfaces in the form of narrow, elongated rectangular pits elongated along  $[110]$ , and faceted on the long sides with practically non-etching planes (111)B and planes (111)A etched by a layered mechanism are faceted in short sides.

In the processes of chemical etching of  $\text{A}^{\text{III}}\text{B}^{\text{V}}$  crystals in electrolyte solutions, the leading role is played by chemisorption of anions by dangling bonds of lattice atoms, which most easily occurs in vacant orbitals of  $\text{A}^{\text{III}}$ -type atoms. In this case, the steps appearing on (111)B surfaces should spread faster than on (111)A surfaces, which causes in case of chemical etching of (001) $\text{A}^{\text{III}}\text{B}^{\text{V}}$  crystals the



**Figure 9.** The positions of the band gaps edges in phosphides and arsenides  $A^{III}B^V$  relative to the Fermi level in gold, the position of the edge of the valence band for room temperature is shown by a blue line, the position of the edge of the valence band for temperature of  $360\text{ }^\circ\text{C}$  is shown by a red line.

appearance of similar-shaped pits but elongated along the direction  $[1\bar{1}0]$ .

The suggested mechanism of the catalytic action of gold on the dissociation of GaAs crystals may also be valid for other crystals  $A^{III}B^V$  with a sphalerite structure if the energy states of the electrons on the dangling bonds of the dimers  $B^V$  on their surfaces allow for the possibility of tunnel exchange with the conduction band of the coating metal. Figure 9 shows the relative positions of the edges of the band gaps of compounds  $A^{III}B^V$  and the Fermi level of gold at room temperature and  $360\text{ }^\circ\text{C}$  (estimates based on data from Ref. [19]).

In this view, the possibility of electron tunneling to gold from states lying in the band gap of both arsenides or near the peaks of their valence band seems obvious.

For GaP and InP, direct experimental or calculated data on the energy positions of lone electron pairs on open bonds of phosphorus dimers existing on P-stabilized crystal surfaces have not been found in the literature known to us. The surfaces (001) of phosphides  $A^{III}B^V$  studied in Ref. [21] by photoelectron spectroscopy were produced using ion etching of crystals followed by vacuum annealing, which made them phosphorus-deficient and dimer-free. Nevertheless, it can be assumed that due to the higher values of negative effective charges on dimerized phosphorus atoms compared with arsenic atoms located in similar positions on the surfaces of arsenide crystals, and the shorter lengths of bridge bonds P–P in phosphorus dimers compared with As–As bonds in arsenic dimers, the energy shift of lone electron pairs relative to the top of valence band of the initial crystal during the formation of dimers for phosphides should be greater than for arsenides. But, apparently, the most significant factor affecting the energy state of the electrons occupying the broken bonds of atoms emerging on the surface of any crystal is the electrophysical state of the medium adjacent to the crystal. When such a medium turns out to be a metal with a surface dipole moment instead of

a vacuum, its interaction with electrons localized on broken bonds of atoms leads to an increase in their potential energy level. In this case, the contribution of this obvious but difficult-to-account factor may become crucial for triggering the processes of catalytic dissociation of phosphides  $A^{III}B^V$  in direct contact with metallic gold.

## 4. Conclusion

This paper substantiates the mechanism of the pronounced catalytic effect of metallic gold on the dissociation of crystals of arsenides and phosphides  $A^{III}B^V$ . The observed anisotropy of the interaction of gold with crystal surfaces and the configurations of the resulting etching pits with the introduction of gold into their volume are explained. The proposed model of the ongoing interphase processes is based on the proximity of the energy positions of the Fermi level for gold and the energy levels of lone pairs of electrons occupying the orbitals of dangling bonds of arsenic or phosphorus dimers on the surfaces of crystals freed from oxides. The tunneling transfer of electrons from such lone pairs to gold creates the prerequisites for the closure of single-electron dangling radical-type bonds that appear in their place to form excited diatomic molecules, the chemical bonds in which finally lose the character of  $sp^3$ -hybridization inherent in interatomic bonds in the initial crystal. Subsequent relaxation processes lead to the formation of a third  $sp$ -hybrid bond between the  $B^V$  atoms and the complete separation of the molecule from the crystal. The pair of  $A^{III}$  atoms remaining on the surface as a result of each such act receives an uncompensated positive charge, which facilitates their transition to the gold lattice with the restoration of the local electroneutrality of the interface. Within the framework of the proposed approach to the processes of gold-induced dissociation of crystals  $A^{III}B^V$  and taking into account the layered nature of etching of densely packed facets, the observed configurations of etching pits filled with metal on various crystal surfaces in contact with gold can easily be explained.

## Acknowledgments

The electron microscopic study was performed using the equipment of the Federal Common Use Centre „Materials Science and Diagnostics in advanced technologies“, supported by the Ministry of Education and Science of Russia.

## Conflict of interest

The authors declare that they have no conflict of interest.

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*Translated by A.Akhtyamov*