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The Deposition of Epitaxial Layers from the Vapour of Organometallic Compounds

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Different methods of depositing epitaxial layers from the vapour of organometallic compounds have been compared. The stability of the deposition processes and new data on the preparation of epitaxial layers of $A^{III}B^{V}$, $A^{II}B^{VI}$, and $A^{IV}B^{VI}$ compounds have been examined.
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I. INTRODUCTION

In the nineteen-twenties, Razuvaev[1] studied the thermal decomposition of organometallic compounds (OMC) in a stream of hydrogen. Later, work by Paneth[2-6] drew attention to alkyl compounds of the transition metals as sources of free radicals in the gas phase at pressures of ~10^{-3} mmHg. Subsequently, Razuvaev and co-workers[7-9] showed that alkyl and aryl OMC act as sources of radicals in the liquid phase. Later, Razuvaev's school studied the mechanism of the decomposition of alkyl and aryl OMC of zinc,[10-12] cadmium,[13-15] mercury,[16-20] germanium,[21-23] tin and lead,[24-32] indium,[33] aluminium,[34-36] and antimony,[37] and a number of other compounds. These studies dealt with both the reactions of the radicals and the formation of metallic phases; by 1960 they had provided a theoretical basis for the study of the deposition of epitaxial layers of binary and multicomponent semiconductors using OMC, an intensively developing field of present-day materials science.

In 1959 it was found that the reaction of trimethylantimony with diborane gives a deposit with the composition BSb_x and gaseous methane.[38] It was subsequently shown[39] that the reaction of trimethylindium with phosphine takes place in three stages:

$$InMe_3 + PH_3 \xrightarrow{-100^\circ C} InMe_3 \cdot PH_3 \xrightarrow{-80^\circ C} (MeInPH)_n \xrightarrow{300^\circ C} InP$$

and that the reactions of trimethylgallium (TMG) with arsine and of trimethylindium with stibine take place according to the schemes:[40]

$$GaMe_3 + AsH_3 \xrightarrow{28^\circ C} \text{grey film} \xrightarrow{200^\circ C} \text{red film} + CH_4$$

$$InMe_3 + SbH_3 \xrightarrow{28^\circ C} (MeInSbH)_n \xrightarrow{160^\circ C} InSb + CH_4 .$$

The practical application of the alkyls of the elements of groups II and III of the periodic system began, however, after the development of the technology of the deposition of epitaxial layers in 1968, when the reaction of TMG with arsine was proposed for the preparation of epitaxial layers of gallium arsenide.[41] In subsequent years, the reactions of the alkyls of the group III elements with ammonia, the hydrides of phosphorus and arsenic, and antimony alkyls were proposed for the preparation of epitaxial layers of the compounds GaAs, GaP, $GaAs_xP_{1-x}$, and $GaAs_xSb_{1-x}$ and the ternary semiconducting compounds $Ga_{1-x}Al_xAs$,[42-45] aluminium phosphides,[46] aluminium and gallium nitrides,[47] indium arsenide and phosphide, and the ternary systems $Ga_xIn_{1-x}As$ and $InAs_xP_{1-x}$,[48] and epitaxial layers of $A^{II}B^{VI}$ compounds.[49,50] The hetero-epitaxial deposition of $A^{II}B^{VI}$ and $A^{III}B^{V}$ layers was reviewed in 1971.[51] Subsequently, the preparation of films by the reactions of alkyls of group IV elements with the hydrides and alkyls of group VI elements ($A^{IV}B^{VI}$ compounds) was described,[52] a number of patents on the preparation of epitaxial structures and hetero-structures by the reactions of OMC with hydrides appeared,[53-56] and the method began to be called the OMC–hydride process†.

Most work has been devoted to the deposition of epitaxial layers of $A^{III}B^{V}$ compounds, because of their widespread use in opto- and micro-electronics. Work on the development of epitaxial $A^{II}B^{VI}$ layers has begun to develop only since 1978, because of the difficulty of obtaining and alloying these compounds. At the same time, the theory of the deposition of epitaxial layers from OMC vapour has been developed, and this is reflected in a number of monographs[57,58] and reviews on the preparation of $A^{III}B^{V}$ [59-63] and $A^{II}B^{VI}$ [64,65] compounds. Except for Ref.65, where data on the thermal decomposition of various OMC in a stream of hydrogen were examined, most reviews have dealt with the preparation of hetero-structures for the production of electronic instruments, and the study of their properties. The present review examines the relationship between the mechanism and kinetics of the reaction and the stability of deposition processes, and discusses the latest data on the preparation of epitaxial layers from the vapour of OMC.

II. COMPARATIVE CHARACTERISTICS OF METHODS OF DEPOSITION FROM THE VAPOUR OF ORGANOMETALLIC COMPOUNDS

Methods of deposition from the vapour of OMC can be classified according to two main characteristics: the compounds used to transport the elements into the reactor, and the deposition conditions. In any method, the deposition from the vapour phase takes place via a number of

†In the foreign literature this method is called the MOCVD method—Metalorganic Chemical Vapour Deposition.

intermediate stages: I) volatilisation of the original compounds, II) mixing and transport of the starting materials, III) diffusion of the vapour of the starting materials to the support, IV) adsorption, V) reaction or thermal decomposition, VI) crystallisation of the epitaxial layer, and VII) removal of gaseous products from the reactor.

Three main methods of transport are used in deposition from the vapour of OMC. Thus binary compounds AB are obtained by the reaction:

$$AR_n(\text{gas}) + BR'_m(\text{gas}) \rightarrow AB(\text{crystalline}) + X(\text{gas}) \qquad (1)$$

where n, m = 1, 2, 3, 4; A is an element from groups II, III, or IV, B an element from groups V or VI, R an alkyl group, R′ a hydrogen atom or an alkyl group, X the gaseous products of reaction (1), and (crystalline) and (gas) denote the condensate and the gas phase. In stages IV and V, reaction (1) can take place with the formation of intermediate polycondensed products with the formula $(-R-A-B-R')_n$, as observed for the reaction of TMG with arsine,[66] so that complexes of the type $AR_n.BR'_m$ are used for the deposition of binary semiconducting structures.[67]

A completely different method of transport involves the use of organobimetallic compounds containing an A–B bond.[68–72] Cadmium sulphide,[68] layers of titanium,[69] and epitaxial layers of $A^{IV}B^{VI}$,[70–72] $A^{II}B^{VI}$, and $A^{III}B^{V}$[73] compounds have been prepared. The use of molecular complexes and organobimetallic compounds makes it possible to reduce the synthesis of a complex semiconducting compound to the preparation of elementary semiconductors, but the stoichiometry and electrical properties of the layers can be controlled only by selecting the temperature and pressure, since the ratio of the reagents A and B is fixed in the original compound.

Depending on the conditions, deposition from the vapour of OMC is carried out by vacuum methods or by methods involving a carrier gas. Historically, methods of vapour-phase deposition based on the transport of the elements or chlorides using hydrogen[74] were transferred, by replacing the chlorides by OMC, to the deposition of epitaxial layers of binary compounds from the vapour of OMC. The process is carried out as follows: the carrier gas, usually hydrogen, is passed through bubblers in which it is saturated by the vapour of the OMC, and then through tubes into the reactor in which the epitaxial layer is deposited. Up to 12 material supply channels are used simultaneously. If a homogeneous reaction between the original compounds is impossible, they are mixed beforehand; if the reaction is possible, they are injected into the reaction zone. The carrier gas is used at atmospheric pressure or at a reduced pressure.[75]

The carrier gas is used as a reference defining the pressure in the deposition system, and makes it possible to transfer precise quantities of the original OMC and to control their supply automatically; this is particularly important in the production of multicomponent structures.

At the same time, the use of a carrier gas has a negative effect on the properties of the layers obtained, because of hydrogen etching of the growing layer, the concentration of impurities from the carrier gas in the epitaxial layer, the transfer of droplets and particles from the volatiliser to the reactor, and the increased difficulty of removing the reaction products. OMC displace light gases from an adsorption layer,[76,77] but since the relative concentration of the reagents does not exceed 1–2 vol.%, the carrier gas nevertheless blocks the adsorption centres, leading to energetic uniformity of the growth surface and hence to a corresponding decrease in the proportion of selective decomposition of the OMC.[78]

Vacuum methods of deposition from the vapour of OMC in the absence of a carrier gas have now been developed.[57,58] This method has been used to obtain epitaxial layers of $A^{IV}B^{VI}$,[72] $A^{III}B^{V}$,[79–82] and $A^{II}B^{VI}$[83–88] compounds. An attempt has been made[89] to obtain GaAs from arsine and TMG vapour under molecular-beam epitaxy conditions; arsine had previously been used as a source of arsenic in molecular-beam epitaxy.[90] In vacuum methods, the purity of the layers obtained is determined by the purity of the OMC and hydrides used, in which the concentrations of metal and non-metal impurities are 10^{-4}–10^{-5} vol.%. Hydrogen purified by diffusion through a palladium membrane contains up to 4×10^{-5} vol.% of oxygen-containing impurities.[91] The proportion δ of impurities in the layer can be determined from the relationship

$$\delta = \frac{j_i}{j_0}\frac{k_i}{k_0}$$

where j_i and j_0 are the fluxes of the molecules of the impurity and the OMC at the growth surface, and k_i and k_0 the coefficients of reactive adhesion of the impurity and the OMC.

The capture of oxygen-containing impurities in an epitaxial layer can be examined for the case of the deposition of $Ga_{1-x}Al_xAs$ from the vapour of TMG, trimethylaluminium (TMA), and arsine.[91,92] The hydrogen stream contained less than 1 vol.% OMC, and comparison of the rates of deposition given by the authors cited and estimation of the fluxes of the OMC into the adsorption layer indicate that for TMG and TMA, the value of k_0 is 1–0.01, whereas $k_i \cong 1$, since the OMC is a compound which is rapidly oxidised. Thus the concentration of aluminium and gallium oxides in the epitaxial layers can apparently reach several tenths of 1%. With decrease in the hydrogen pressure, therefore, layers of high quality are obtained at lower deposition temperatures than at atmospheric pressure.[75] Decrease in pressure facilitates the effective removal of the gallium oxide decomposition products from the reaction zone. This is confirmed by data[92] which show that at a concentration of oxygen-containing impurities in the hydrogen stream at the level of 10^{-6} vol.%, the $Ga_{1-x}Al_xAs$ layers with $x < 0.05$ were of high quality, whereas at $x > 0.05$ the conductivity and photoluminescence yield of the layers decreased sharply, since aluminium oxides are stable under the deposition conditions.

The thickness of the oxide on the $Ga_{1-x}Al_xAs$ surface is proportional to the value of x,[93] and layers of high quality are obtained only after the hydrogen has been specially purified from oxygen-containing impurities.[91] In deposition in a vacuum, the purity of the layers is determined by the purity of the OMC and the ratio of the flux corresponding to the leakage from the atmosphere into the reactor to the flux of the starting materials into the reactor. At a level of leakage into the reactor lower than the sensitivity of the helium leak detector, and fluxes of the original reagents into the reactor of ~10 litre mmHg s^{-1}, the concentration of oxygen-containing impurities due to leakage into the reactor is not more than 10^{-9} vol.% (without allowance for the absorption of oxygen and water vapour by the walls of the reactor and by the reaction mixture).[79]

In deposition under the conditions of molecular-beam epitaxy, the pressure of the residual gases in the reactor is 10^{-10} mmHg, and the fluxes of the atoms on to the growth surface corresponds to pressures of 10^{-5} mmHg, so that the purity of the layer with respect to the impurities present in the residual gases is 10^{-3} vol.%. The use of improved evacuation techniques will be unlikely to lower significantly the level of contamination of layers obtained in this way due

to residual gases, since at low pressures of the original reagents in the reactor, the atomically pure surface of the growing crystal actively captures impurities.

The mechanisms of the reactions taking place in the adsorption layer depend on its density and the quantity F, equal to the ratio of the energy of intermolecular interaction in the adsorption layer to the energy of the interaction of the adsorbed molecules with the surface. At a low density of molecules in the adsorption layer, $F \ll 1$, the original reagents react with the surface of the support, and a vapour–crystal (VC) growth mechanism is realised. At a medium density of the adsorption layer and a value of $F \cong 1$, the formation of surface compounds which can play the role of liquid is possible, and in addition to the VC growth mechanism, the vapour–liquid–solid (VLS) growth mechanism may be observed. At a high density of the adsorption layer, the value of $F \gg 1$; growth of the layer takes place by the VLS mechanism, drops of liquid are generated from the original elements on the surface, and metastable phases, polymer chains containing metal, carbon, and hydrogen, may be formed.

These relationships can be illustrated by the reaction of TMG with arsine. The reaction of TMG with arsine takes place via the formation at 203° of co-condensation products Me_2–Ga–As–H_2, which at 259° decompose with the liberation of condensation products $(Me–Ga–As–H)_n$, which subsequently decompose at 420 °C with the liberation of gallium arsenide.[66] A study of the deposition of gallium arsenide from arsine and TMG vapour at a hydrogen pressure of 75 mmHg[75] showed that an amorphous deposit with a thickness of up to 500 Å remains on the surface of the epitaxial layer; the photoluminescence spectrum of this deposit showed a weak broadened band at 1.37 eV, and the edge band appeared only after this surface amorphous layer had been etched; the layer was apparently formed by residues of the co-condensation products of TMG and arsine, involved in the growth of gallium arsenide. In the deposition of gallium arsenide in a vacuum,[80] at Knudsen numbers (Kn) in the flux of arsine and TMG vapour of 1–0.1, no residual products were observed on the surface of the films. At Kn ≅ 1 and a deposition temperature of 400°, block single-crystal layers were obtained; the epitaxy temperature was 430°, as in deposition by the method of molecular-beam epitaxy; at temperatures above 500°, highly perfect layers were obtained. At Kn ≅ 0.1, the epitaxy temperature increased to 500°, corresponding to the regular features observed in deposition in a stream of hydrogen at a reduced pressure.

A study of the defects in gallium arsenide layers obtained by deposition in a vacuum[82] showed that the films deposited at Kn ≅ 1 had a rough surface, on which drops with dimensions of up to 300 Å were observed; the principal defects were dislocations, twins, and subtractive packing defects. As Kn → 0, the size of the droplets increased to 1500 Å with an increase in the density of defects, followed by a sharp change to the growth of layers with an atomically smooth surface and minimum density of defects, in spite of the fact that the concentration of p-type carriers in the film reached 10^{19}–10^{20} cm^{-3}. This change in the properties of the layer with increase in the pressure at which the deposition was carried out can be attributed to a change in the growth mechanism from VC to VLS; the agent forming the liquid on the surface of the growing layer may be the intermediate compound considered above.

The characteristic features of the alloying of gallium arsenide with selenium are also related to the mechanism of the reaction in the adsorption layer. Thus when hydrogen selenide is introduced into the stream,[82] highly compensated specimens of n-type gallium arsenide are obtained, and the morphology of the surface and the concentration of defects correspond to the picture observed in the growth of layers with an excess of TMG, that is the introduction of a small quantity of selenium into the adsorption layer facilitates the "attraction" of TMG molecules, revealed in the apparent displacement of the conductivity conversion point. These data agree with the results of published work[94] where displacement of the conductivity conversion point was observed in the deposition of gallium arsenide from TMG and arsine with change in the concentration of impurities in the TMG.

The purity of binary semiconducting layers is determined to a large extent by the stoichiometry, since impurities are captured preferentially on the vacancies. This is illustrated by the deposition of layers of zinc selenide by the reaction of diethylzinc (DEZ) and hydrogen selenide.[85,88] Small quantities of ethyl iodide were introduced into the DEZ to a concentration of 0.01–1 vol.%. During the deposition, the temperature of the support was 350°, and the pressure in the reactor 0.2 mmHg. Zinc selenide was deposited by the reaction

$$ZnEt_2 + H_2Se \rightarrow ZnSe + 2EtH \qquad (2)$$

accompanied by the thermal decomposition of DEZ, which takes place vigorously at temperatures above 300°,[95,96] while zinc reacts with hydrogen sulphide according to the equation

$$Zn + H_2Se \rightarrow ZnSe + H_2 .$$

In the presence of an alkyl iodide, however, the zinc atoms can react with it to form the intermediate compound Et–Zn–I,[97] which undergoes symmetrisation to give DEZ and zinc iodide; the synthesis of DEZ from zinc and an alkyl iodide takes place up to 380°,[98] and the symmetrisation of the intermediate compound EtZnI is the limiting stage.[99]

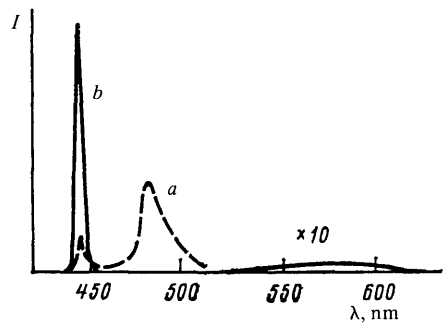


Figure 1. Photoluminescence spectra of zinc selenide layers; ratio of the fluxes of DEZ and hydrogen selenide 1 : 2 (*a*) or 1 : 5 (*b*).

Reaction (2) is rapid, so that the thermal decomposition of DEZ is possible only when there is an excess of it in the adsorption layer;[93] in this case, in the presence of an alkyl iodide, the compound EtZnI is formed and then decomposes to give Zn–I· radicals. The condensation of this radical leads to the capture of an iodine atom on a selenium vacancy in zinc selenide. Thus the photoluminescence spectrum of zinc selenide specimens obtained at a ratio of the DEZ flux to the hydrogen selenide flux into the reactor of 1 : 2 (Fig.1*a*), in addition to the edge band, shows a peak

at 480 nm, corresponding to an iodine donor level, and with change in the ratio of the fluxes of DEZ and hydrogen selenide to 1:5, the intensity of this peak decreases by a factor of 300[88] (Fig.1*b*). Thus the capture of the iodine impurity is due to the thermal decomposition of DEZ and the formation of EtZnI intermediate compounds, and the observed displacement of the iodine impurity by the growing crystal front is due to the suppression of the synthesis of the compound containing the Zn–I bond. The characteristics of the methods of molecular-beam epitaxy, deposition from OMC vapour in a vacuum, and the OMC–hydride process in a stream of hydrogen are compared in Table 1.

Table 1. Comparison of the characteristics of the methods of molecular-beam epitaxy and the OMC–hydride process in a stream of hydrogen and in the absence of a carrier gas.

Method	γ*	Desorption	LSC**	Growth mechanism	Deposition conditions
Molecular-beam epitaxy	10^{-5}	fast	attainable	VC	supersaturation rigidly related to the flux; removal of products not limited by diffusion
OMC-hydride process in hydrogen	10^{-7}–10^{-2}	blocked	not attainable	VLS	supersaturation depends on the reagent fluxes; removal of products limited by diffusion
OMC-hydride process without a carrier gas	up to 10^{-10}	fast	attainable	VC or VLS	supersaturation depends on the reagent fluxes and the pressure in the reactor; removal of products not limited by diffusion

*Ratio of the sum of the fluxes due to leakage into the reactor, residual gases, and impurities in the carrier gas to the flux of the original reagents reaching the support.
**Local stability of the concentrations.

The differences between the epitaxy processes in a vacuum and in a stream of carrier gas are considerable, particularly in stages III, IV, and VII. The carrier gas, which brings about the transport of the OMC from the volatilisers into the reactor, simultaneously acts as measurement reference for determining the pressures of the components in the stream. For deposition in a vacuum, this reference is not available, and this creates a number of difficulties in the control of the fluxes and the stabilisation of the deposition conditions, and requires examination of the dynamic process as a whole.[57,58]

The gas-dynamic conditions in the reactor when a carrier gas is used are traditionally studied by introducing an aerosol into the gas stream;[100] the aerosol particles, however, do not detect the fine swirling movements which are detected by interferometry. The gas dynamics of reactors of different design have been studied using a Mach–Zehnder interferometer.[101] The results of the study of the gas dynamics of horizontal reactors have been reviewed.[102] The relationship between the development of the growth surface and gas-dynamic factors has been studied.[103–105]

At pressures below 20 mmHg, schlieren and interferometric methods are not effective, so that indirect methods have to be used to study the flow of the vapour on to the support. The simplest method is to study the profile of the deposited layer, or to record the temperature and pressure profiles in the vapour stream. The reaction of DEZ with hydrogen selenide is used to make visible the flow picture at pressures of $10–10^{-3}$ mmHg.[106] The layer of zinc selenide deposited on a probing plate makes it possible to obtain a detailed picture of the flow on to the support from the tarnish iridescence colours, and the luminescence of the layer can be used to study the mixing of the streams when they are injected into the reactor and the separation into layers of components with different molecular weights.

The vacuum systems for epitaxy from OMC vapour consist of four main units: the volatiliser, the reactor, the traps, and the evacuation system; they operate in the pressure range 10^{-2}–5 mmHg, where viscous gas flow conditions are replaced by molecular flow conditions, and are characterised by a high degree of conversion of the original reagents. The diffusion of the OMC to the support takes place through a layer of reaction products diffusing in the opposite direction, and a complex relationship is established between the reaction rate, the pressure in the reactor, and the thickness of the "reduced" film. The rate of evacuation of the products from the reactor, the rate of the supply of the OMC vapour to the reactor, and the degree of transformation in the reactor are also related in complex fashion. Overall, however, the process of deposition from OMC vapour in a vacuum is autonomous.

The theory of dynamic vacuum systems for deposition from OMC vapour has developed comparatively recently, since 1967. Thus the fluctuations in the growth rate as a result of competition between stages III and VII have been examined.[107] According to the proposed model, binary diffusion of the OMC and the thermal decomposition products takes place in the "reduced" film, and the rate of removal of the reaction products from the reactor is of the same order of magnitude as the rate of binary diffusion. In this case, fluctuations in the concentrations of the OMC and the decomposition products can take place in the reactor, and the frequency of the fluctuations is related to the reaction rate constant, the rate of evacuation, and the geometric parameters of the reactor. Analysis of the model for stability of the stationary deposition conditions showed that the higher the rate of pumping and the smaller the mass-transfer constant in the reduced film, the higher the probability that fluctuational conditions will be established; the frequency of the fluctuations increases with increase in the rate of pumping, and decreases with increase in the mass-transfer coefficient to the surface of the support. The fluctuations in the concentrations of the OMC and the thermal decomposition products lead to non-uniformity of the composition of the film along its thickness and to the growth of layer structures.

A model of acoustic self-fluctuations in the density of the vapour in the reactor, brought about by the transfer process at the start of the deposition, has been examined.[108] In the approximation of a differential reactor, with simplifying assumptions about the pressure and temperature profiles, it was shown for the case of viscous flow in a long cylindrical reactor that the acoustic wave produced when the OMC vapour is introduced into the reactor is reflected, returns to the support, and induces a change in the OMC concentration at the support. At certain ratios of the reaction rates, the geometric parameters of the reactor, and the vapour pressure in the system, sustained fluctuations of the density are excited, and their energy is supplemented by the liberation of gas during the thermal decomposition.

The self-fluctuations in the rate of growth of a film due to thermal phenomena such as the finite nature of the rate of heat transfer from the heater to the surface of the support

and the absorption or liberation of heat in the reaction on the surface have been studied.[109] If the thermal decomposition of the OMC is endothermic and autocatalytic, the liberation of solid products leads to an increase in the reaction rate, an increase in the rate of removal of heat, and a decrease in the temperature of the support; as a result, the degree of coverage of the surface with thermal decomposition products increases, the reaction rate decreases, the temperature of the surface increases, and the cycle is repeated. If the decomposition of the OMC is kinetically restricted (at low temperatures), self-fluctuations in the temperature of the support will be excited if the power of the heater is insufficient or if the support has a low thermal conductivity. The change to diffusion deposition conditions suppresses the self-fluctuations. Exothermic reactions also lead to the excitation of fluctuations in the temperature of the support, since the liberation of heat in the reaction leads to an increase in its rate and to "depletion" of the starting materials, the reaction rate decreases, the temperature of the surface decreases, and the cycle is repeated. A low thermal conductivity of the support leads to an increase in the amplitude of the fluctuations; the heat liberated in the reaction supplements the energy of the fluctuations.[58]

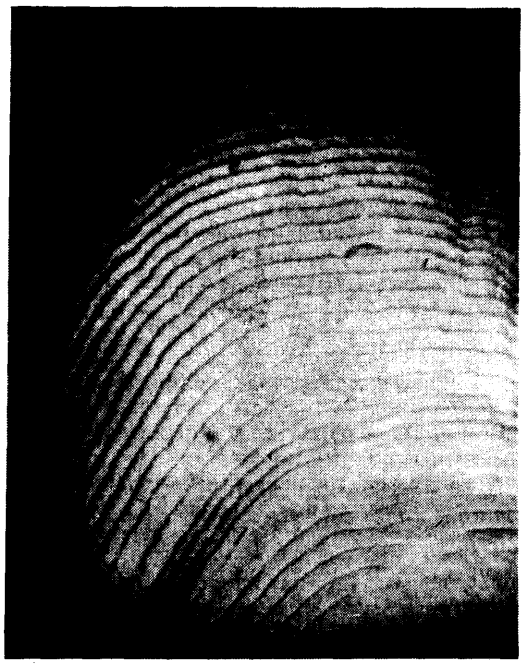


Figure 2. Transverse section of a layer of zinc selenide. Dark layers—with an excess of selenium; light layers—stoichiometric zinc selenide.[85]

The fluctuations in the pressure in the reactor and the corresponding changes in the composition of the layer being deposited have been observed experimentally; the experimental results agreed with the theory.[110,111] Essentially, all the above models relate to cases where competition between various types of mass transfer in the deposition system (stages II, III, and VII) moves the reaction from kinetic conditions to diffusion conditions or vice versa; this is a characteristic feature of deposition from OMC vapour in a vacuum, since at pressures of ~0.1 mmHg the concentrations of the OMC in the volume ($\sim 10^{15}$ cm^{-3}) and on the surface ($\sim 10^{15}$ cm^{-2}) are of the same order of magnitude. The fluctuations are suppressed when the deposition conditions move "deep" into the diffusion range, but this is not always possible, because of the influence of pressure and temperature on the quality of the deposited layer.

The fluctuations in pressure in the volatiliser–reactor–trap system, arising in the production of a binary compound AB by several parallel reactions, when the rate of the leading reaction is much greater than those of the other reactions, and when hydrogen, which is not frozen out in the trap, is liberated in the reaction products, have been examined.[112] The products of the main reaction are condensed in the trap. The highest rate of deposition corresponds to the growth of the stoichiometric layer AB, the graph giving the pressure dependence of the growth rate passes through a maximum, and fluctuations of pressure in the reactor should be excited at a certain critical pressure and are accompanied by fluctuations in the rate of liberation of hydrogen. Fluctuational conditions of this kind were observed in the preparation of zinc selenide by the reaction of DEZ with hydrogen selenide.[85] The fluctuations in pressures were accompanied by the growth of a structure consisting of periodically alternating layers of stoichiometric zinc selenide (light layers in Fig.2) and ZnSe with an excess of selenium (dark layers in Fig.2). The frequency of the fluctuations was related to the volume of the reactor, the working pressure, and the rate of removal of the volatile component.[112]

The fluctuations in the concentrations of the components of the reaction in the adsorption layer, due to the characteristic features of the desorption and adsorption of the OMC and the phenomena of heterogeneous catalysis and development of the growth surface, have been examined.[58,113] The fluctuating change in the surface concentrations in the adsorption layer is excited by autocatalytic phenomena, the activated chemisorption of the OMC and the decomposition products, the multistage nature of the surface reaction, and the two-dimensional condensation of intermediate phases, and can take place without a change in the pressure in the reactor.

Thus methods of deposition in a carrier-gas stream have been developed technically, and make it possible to produce multicomponent mixtures of OMC and hydrides under dynamic conditions, automatically, and with a high accuracy. A high carrier-gas pressure in the deposition system makes it possible to separate stages I and II, III and VII. The quality of the layers obtained by this method is decreased by contamination by impurities from the carrier gas, etching of the growing layer, blocking of the adsorption centres by molecules of the carrier gas, the consequent decrease in the fraction of the selective decomposition of the OMC, and local instability of the concentrations, due to convective and turbulent diffusion and small-scale rotational movements in the boundary layer.

The vacuum method of deposition from OMC vapour does not have the disadvantages associated with the use of a carrier gas, and this makes it possible to improve considerably the quality of the layers and to achieve automatic control of the deposition process, finer control of the supersaturation, and stabilisation of the fluxes of the reagents to the growth surface. This method is more complicated, however, in the generation of the fluxes of the original OMC, and requires detailed experimental and design studies to

find stable deposition conditions; this is related to the use of flow conditions close to the range corresponding to the change from viscous to molecular conditions.

III. DEPOSITION OF EPITAXIAL LAYERS OF $A^{III}B^{V}$ COMPOUNDS

Epitaxial layers of binary semiconducting compounds $A^{III}B^{V}$ are obtained by the reactions of OMC and hydrides and molecular complexes. There is comparatively little published information on the mechanism of the reactions accompanying deposition. A study of the reaction of TMG with arsine[66] (see above) showed that the polycondensed fragments containing the $\rangle$Ga–As$\langle$ bond undergo autocatalytic decomposition to gallium arsenide. The homogeneous nucleation of aggregates $(GaAs)_n$,[114–116] the formation of homogeneous polymeric species containing Ga, N, C, and H atoms in the reaction of TMG with ammonia, and the formation of polymeric layers containing In, P, C, and H in the reaction of trimethylindium with phosphine[117] have been proposed, but no direct proof of homogeneous nucleation has been give.

The conditions for the distillation of the molecular complexes $ClEt_2Ga.AsEt_3$, $ClEt_2Ga.AsMe_3$, $ClEt_2Ga.NEt_3$, and $ClEt_2Ga.PEt_3$,[118] which can be used to obtain layers of $A^{III}B^{V}$ compounds, have been studied. The complex $Me_3Al.NH_3$ has been used to obtain aluminium nitride.[47] The first methane molecule is removed at 60°, and the second at 160°; decomposition to form AlN is complete at 1200°.[47,119,120] The thermal decomposition of the complexes $Cl_3Al.NH_3$ and $Me_3Ga.NH_3$ [121,122] and the decomposition of the complexes $Et_3Ga.NH_3$, $R_3Ga.AsR_3$,[123] $Me_3In.PMe_3$,[124] and $ClEt_2Ga.AsEt_3$ [125] have been studied. A number of complexes containing the Ga–As bond have been studied to obtain layers in a horizontal flow reactor in a stream of helium.[126] It was reported that the type of conductivity depends on the method used to heat the support; the authors cited consider that this was proved by the fact that the Ga–As bond is not broken, although change in the type of conductivity is due rather to a change in the type of self-alloying impurities when working with a hot or cold reactor wall. The complex $(Et_2Ga.PEt_2)_3$ has been used to obtain layers of GaP.[127] The thermal decomposition of $ClEt_2Ga.AsEt_3$ has been studied.[128] The deposition of the ternary system $GaAs_{1-x}P_x$ with values of x in the range 0–0.6 from $ClEt_2Ga.AsEt_3$ and diethylphosphine vapour has been examined.[129]

The process of deposition from OMC vapour is analysed by the method of partial pressures,[130] which involves the numerical solution of systems of equations for the partial pressures and equilibrium constants, and the subsequent construction of equilibrium diagrams on coordinates temperature–pressure–composition. These diagrams have been constructed for the system Ga–As–Cl–H; a comparison[131] of the deposition of gallium arsenide from OMC vapour with the chloride–hydride process showed that the OMC–hydride process has a wider range of conditions for the existence of gallium arsenide, makes possible a smoother control of the supersaturation, and is less critical with respect to the degree of stabilisation of the conditions of the experiment. A study of GaAs layers deposited from a mixture of the vapours of arsine and the ether addition compound of TMG, labelled in the methyl group with the ^{14}C isotope, showed that the ^{14}C concentration did not exceed 6×10^{13} cm^{-3} and agreed with the thermodynamic analysis.[132,133] A thermodynamic analysis of the system TMA–trimethylantimony showed that at the deposition temperature of aluminium antimonide (623°) the product is aluminium carbide, and that the use of tri-isobutylaluminium instead of TMA makes it possible to decrease sharply the concentration range corresponding to the synthesis of aluminium carbide,[134] and this was confirmed experimentally.[135] Subsequent work was directed towards the preparation of the ternary and quaternary systems $Ga_{1-x}Al_xSb$ and $Ga_{1-x}Al_xAs_{1-y}Sb_y$ [136] and the study of the influence of the pressure of the OMC of group III elements relative to the vapour pressure of the compounds of group V elements on the composition of the $GaAs_{1-x}Sb_x$ layers obtained; the results of the thermodynamic calculations agreed with the experimental data.[137,138]

Published data on the kinetics of the growth of $A^{III}B^{V}$ epitaxial layers from OMC vapour are obtained by recording the dependence of the rate of deposition on the parameters of the process. The rate of deposition is independent of temperature in the range 600–800°. *..... semiconducting compounds and the development of instruments based on epitaxial layers, *..... group V, and is of first order with respect to the consumption of the OMC vapour. The process is generally carried out under conditions of diffusion control, so that the rate of deposition increases with increase in the flux rate.[41,75,139]

The mechanism of the deposition of gallium arsenide by the reaction of TMG with arsine has been studied by infrared spectroscopy. Thus TMG begins to decompose in a stream of hydrogen at a temperature above 400°, and the rate of the thermal decomposition is stabilised above 600°. Arsine begins to decompose at 650°; above 750°, the degree of conversion of AsH_3 is close to 1. When TMG, arsine, and hydrogen are introduced into the reactor, the infrared spectrum of the products shows a new peak at 2080 cm^{-1}, whose intensity increases with increase in the concentration of arsine in the stream and is a maximum in the temperature range 510–550°.[140]

Most work on the deposition of layers of $A^{III}B^{V}$ compounds from OMC vapour in recent years has been carried out in two main directions: the preparation and improvement of the properties of ternary and quaternary semiconducting compounds and the development of instruments based on epitaxial layers deposited from OMC vapour. Considerable progress has been made in these directions. Much work has been devoted to the production of lasers based on the hetero-structure $Ga_{1-x}Al_xAs$; lasers based on binary hetero-structures which operate for a long time at room temperatures, highly efficient solar batteries and batteries operating with the concentration of solar radiation,[141–144] field transistors,[145] photocathodes,[146] super-high-frequency transistors with record low noise values,[147] and a number of instruments for opto- and micro-electronics have been developed. The preparation of the quaternary system $Ga_{1-x}In_xAs_{1-y}P_y$ by the reaction of alkyl OMC with the hydrides of the group V elements with the preliminary decomposition of phosphine has been studied,[148] and this has led to the development of highly efficient semiconducting lasers in the near infrared range.[149] The ternary system $Ga_{1-x}In_xAs$ has been deposited,[150] and used to produce a number of opto-electronic instruments.[151] The systems $GaAs_{1-x}Sb_x$ [152] and $In_{1-x}Ga_xP$ with $x = 0.5$ [153] have also been prepared.

Work is continuing on the hetero-epitaxy of aluminium nitride from a mixture of the vapours of OMC, ammonia, and hydrazine on supports of silicon and sapphire for acoustic detectors.[154–156] Lasers based on $Ga_{1-x}Al_xAs$,

*Misprint in Russian text: two or more lines missing.

stable in proton fluxes,[157] layers of aluminium arsenide,[158] solid solutions $Ga_{1-x}Al_xN$,[159] and films of gallium arsenide on molybdenum[160] have also been obtained.

Intensive studies aimed at lowering the deposition temperature are being carried out[161-163] to produce epitaxial layers of high quality. The greatest success in this direction has been achieved by carrying out the reaction of TMG with arsine in a glow-discharge plasma. Thus the reaction of TMG with arsine at room temperature in the plasma of a helium discharge gave layers from which GaAs was isolated by treating with an electron beam.[164] A process has been developed for preparing a GaAs surface in the plasma of a helium discharge at 200° with the addition of HCl,[165] and layers of gallium arsenide suitable for use in instruments have been obtained.[166] The most perfect layers wer obtained at a support temperature of 400–450° and a pressure of helium vapour in the reactor of 1.5 mmHg.

Deposition from OMC vapour makes it possible to obtain sharp boundaries between layers with different compositions. Thus a superlattice has been produced[167] by alternating layers of n-$Ga_{1-x}Al_xAs$ (240 Å) with layers of unalloyed GaAs (225 Å). Study of the alloying profile in the layer showed that the lattice period (the distance between successive carrier concentration peaks) is ~250 Å, and that the mobility of the carriers is greater than the theoretical value; this opens up the possibility of producing improved devices.

Thus $A^{III}B^{V}$ epitaxial layers are obtained by deposition from OMC vapour using a wide range of compounds which ensure relatively low deposition temperatures and controlled alloy production. The use of OMC makes it possible to achieve precise control of the supersaturation, to obtain multicomponent systems, and to ensure that the parameters of the layer are constant over a wide area.

IV. DEPOSITION OF EPITAXIAL LAYERS OF $A^{II}B^{VI}$ AND $A^{IV}B^{VI}$ COMPOUNDS

Epitaxial layers of $A^{II}B^{VI}$ compounds show extensive promising possibilities for application in opto-electronic and electroluminescent instruments in the visible range of the spectrum.[168] Considerable effort has been put into the production of $A^{II}B^{VI}$ films, but success has been achieved only in recent years, when deposition from OMC vapour was used for this purpose. The ability of $A^{II}B^{VI}$ compounds to undergo self-compensation makes it possible to obtain conducting layers only with a low density of point defects, so that the deposition process presents more rigorous requirements regarding the level of purity from oxygen-containing impurities and the accuracy of the control of the supersaturation, compared with $A^{III}B^{V}$ compounds. Epitaxial layers of the chalcogenides of the group II elements are obtained from OMC vapour using reaction (1), and the reagents used are dimethylcadmium, dimethylzinc (DMZ), DEZ, sulphur and selenium hydrides, selenium and tellurium alkyls,[57,58] and heterocyclic derivatives of sulphur, selenium, and tellurium.[169] The thermal decomposition of the alkyls of the group II elements zinc[9-11] and cadmium[12-14] has been studied.

Table 2 gives published data[170-174] on the kinetics of the thermal decomposition of alkyls of the elements of groups II and VI. The thermal decomposition of DEZ and DMZ has been studied under static conditions,[170] in a stream of helium,[171] and in a high vacuum.[172] The decomposition of a mixture of DEZ and dimethylselenium has been studied.[173] In the decomposition of DEZ in a vacuum at a pressure of 10^{-5} mmHg, the following gaseous products were detected: CH_4, C_2H_6, C_2H_4, C_4H_8, and C_4H_{10}. Increase in temperature led to an increase in the yield of hydrogen, ethylene, and butene, and a decrease in the yield of ethane.[172] In the decomposition of DEZ under static conditions, the reaction was homogeneous. In addition to the gaseous products listed above, octane, octene, hexane, hexene, butane, and butene were detected. The data in Table 2 show that the activation energy increases with increase in the pressure and the proportion of the homogeneous reaction. Fig.3 gives published data[65] on the thermal decomposition of alkyls of cadmium, tellurium, and selenium in a stream of hydrogen. The temperature dependence of the deposition rate is determined entirely by the mass-transfer constants, as can be seen by comparing the curves for the decomposition of diethyltellurium (Fig.3). In the thermal decomposition of a mixture of DEZ and dimethylselenium, the limiting stage is the decomposition of dimethylselenium, and the ethyl radicals formed by the decomposition of DEZ induce the decomposition of $SeMe_2$.[173,174]

Table 2. Decomposition of zinc and selenium alkyls.

Compound	T, °C	Decomposition conditions	$\lg k = A - E_a/4.58\,T$: E_a, kcal mole^{-1}	A	Refs.
$ZnMe_2$	287—348	S.C.*	35.5	9.63	[170]
	250	flow, 10^{-5} mmHg	31.6	—	[172]
	230	flow, 10^{-4} mmHg	—	—	[171]
$ZnEt_2$	270—310	S.C., 10–100 mmHg	28.3	8.44	[171]
	310—340	flow, 760 mmHg	47.6	16.13	[171]
	100—300	flow, 10^{-5} mmHg	24.4	—	[172]
$ZnEt_2 + SeMe_2$	350—400	S.C., 90–330 mmHg	30.5	7.23	[173]
	350—400	flow, 760 mmHg	20.07	3.93	[174]

*Static conditions.

A method has been developed for obtaining dynamic P–T diagrams for heterogeneous OMC–support systems.[95,96] For a dynamic flow reactor a line is constructed on coordinates P, T, along which the rate of deposition of W according to the reactions

$$\mathrm{OMC(gas)} \rightleftarrows \mathrm{M(crystalline)} + \mathrm{X(gas)} \qquad (3)$$

$$\mathrm{M(crystalline)} \rightleftarrows \mathrm{M(gas)} \qquad (4)$$

is equal to zero after allowance for the volatilisation of the metal. Here, M is the metal, and X the gaseous products. An expression has been obtained[175] for the rate of deposition for reaction (3), assuming that it takes place in three stages (III, IV, V), without allowance for volatilisation of the metal. Using the published data[175] and introducing the symbols P_{OMC} and P^0_{OMC} for the vapour pressures of the OMC close to the support and in the flow, and P_X and P_M for the pressures of the reaction products X and the metal vapour close to the support, it is possible to write the condition fulfilled on the line $W = 0$ as follows:

$$k_1 P_{OMC} - k_{-1} P_X + \alpha P_M = \alpha\gamma P^0_M \qquad (5)$$

where k_1 and k_{-1} are the rate constants of the forward and back reactions (3), α the coefficient of condensation of the metal, P^0_M the equilibrium vapour pressure of the metal, $\gamma = P^{cr}/P^0_M$, and P^{cr} the critical supersaturation required for condensation of the metal on the support. For example, for the condensation of zinc on glass, $\gamma = 3$.[176] Condition (5) for $W = 0$ for a glass reactor, with allowance for the volatilisation of the metal, then has the form:

$$P^0_{OMC} = \frac{3\alpha P^0_M k_d}{\alpha + k_d}\left(\frac{1}{k_1} + \frac{1}{k_d} + \frac{1}{K \cdot k_d}\right)$$

where K is the equilibrium constant of reaction (3), and k_d the mass-transfer coefficient in the reduced film (for simplicity, it is assumed that the values of the transfer constants for OMC, M, and X are the same). At low pressures, the thickness of the reduced film is much greater than the dimensions of the reactor, and the following relationship is fulfilled on the line $W = 0$

$$\frac{R}{k_d} = \frac{\alpha\gamma k_d P^0_M}{\alpha + k_d}\left(\frac{1}{k_1} + \frac{1}{k_d} + \frac{1}{K \cdot k_d}\right) \qquad (6)$$

where R is the consumption of the OMC. It is possible to select the temperature and pressure in the reactor in such a way that the conditions $\alpha \gg k_d$ and $k_d \gg K$ are fulfilled. Eqn.(6) then takes the form:

$$\frac{R}{k_d^2} = \frac{\gamma P^0_M}{k_1} .$$

This relationship makes it possible to determine the activation energy of reaction (3). For the case of the volatilisation of the layer from the support, condition (5) takes the form:

$$k_1 P_{OMC} - k_{-1} P_X = \alpha P^0_M . \qquad (7)$$

γ can be determined by comparing the temperatures of the volatilisation and condensation of the layer of metal and using Eqns.(5) and (7).

A detailed analysis of the influence of the conditions of the experiment on the observed activation energies of reaction (3) (without allowance for the volatilisation of the metal) has been carried out.[175]

The kinetics of the deposition of $A^{II}B^{VI}$ epitaxial layers have been studied.[177–182] The following method was used: the vapour of the alkyl of the group VI element was passed through a preliminary reactor (temperature 800°), in which the alkyl of the group VI element was converted into the corresponding hydride in a stream of hydrogen. The mixture of hydrogen, the hydride, and the decomposition products of the alkyl radicals was passed into a reactor in which the chalcogen hydride reacted with the OMC vapour. Cadmium and zinc sulphides and selenides were obtained in this way. The use of the preliminary reactor leads to the loss of the main advantage of the alkyls of the group VI elements over the hydrides, namely the possibility of mixing beforehand with OMC vapour.

Concentration of carbon in the specimens lay in the range $(4–8) \times 10^{20}$ cm^{-3} [177] or $(1–5) \times 10^{20}$ cm^{-3}.[178] The high concentration of carbon in the layers is apparently due to the capture by the growing layer of the cracking products of the alkyl radicals, and depends little on the deposition temperature. In fact, the concentration of carbon in the layers changes in the opposite sense to the rate of deposition.[178] The temperature dependence of the deposition rate shows two peaks: one at about 200°, and the second at about 550°.[177–179] Fig.4 gives the temperature dependence of the growth rate for the deposition of cadmium sulphide from a mixture of OMC vapour[177] and for condensation from a mixture of cadmium and sulphur vapour.[181] The regular features are similar; the curve obtained for condensation from the elements is displaced towards higher temperatures, because of the greater supersaturation, and in the deposition from the vapour of the OMC and the hydride, a decrease in the deposition rate is observed in the low-temperature range.

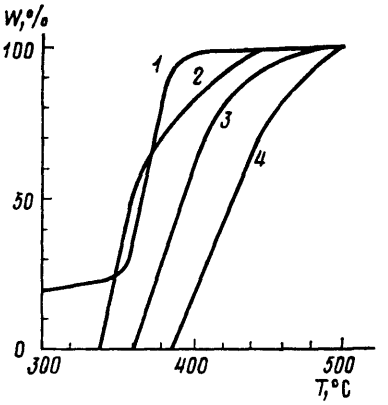


Figure 3. Thermal decomposition of cadmium and tellurium alkyls;[65] W is the ratio of the decomposition rate to the maximum decomposition rate. Compounds: $CdMe_2$ (*1*), $TeEt_2$ (*2*, *3*), $TeMe_2$ (*4*). Pressure of the OMC vapour in the stream of hydrogen: 9 mmHg (*1*), 2 mmHg (*2*, *3*), 10 mmHg (*4*); hydrogen flow velocity: 20 cm s^{-1} (*1*, *3*), 10 cm s^{-1} (*2*), 15.6 cm s^{-1} (*4*).

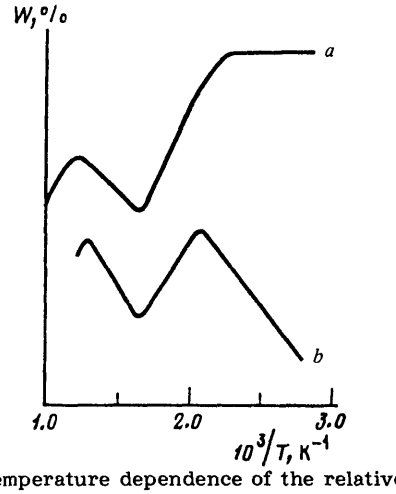


Figure 4. Temperature dependence of the relative rate of growth of cadmium sulphide: *a*) condensation from cadmium and sulphur vapour,[181] *b*) deposition from the vapour of $CdMe_2$ and the decomposition products of dimethyl sulphide.[177]

Up to 1 at.% gallium was detected in layers deposited on gallium arsenide supports; it was assumed[179] that this is transferred to the layer from the opposite side of the support. A mechanism has been proposed[179] for the growth of CdS layers from dimethylcadmium vapour and

hydrogen sulphide. It is suggested that growth takes place by a VLS mechanism with the formation of a two-dimensional liquid from cadmium atoms on the CdS surface. The most perfect layers were obtained with an excess of dimethylcadmium in the stream. Zinc selenide has been obtained by the reaction of DMZ with dimethylselenium in a stream of hydrogen.[180] The plot of the growth rate against the reciprocal of the temperature had a maximum at 560°, and at lower deposition temperatures, W decreased linearly with increase in $1/T$. H_2Se and methane were detected in the gaseous products. The cathodoluminescence spectra of the layers showed an intense band at 600 nm, a number of bands in the range 500–460 nm, and an edge band with phonon structure, indicating that the ZnSe layers obtained had a non-stoichiometric composition and a high density of defects.

The growth rate of a layer of a compound AB by reaction (1) is a function of the pressure in the reactor and the ratio of the fluxes of the OMC and the hydride in the reactor β.[87] Change in the deposition temperature as a result of a difference in the thermal stabilities of the OMC and the hydrides will lead to a change in the ratio β and the pressure in the reactor, and the recording of the temperature dependence of the growth rate may pass through different sections $W(\beta, P)$. For the deposition of ZnSe from the vapour of the OMC and the hydride, the reaction rate over a wide range of conditions is limited by stages II and VII. With increase in temperature and pressure in the reactor, the homogeneous synthesis of ZnSe begins (5 mmHg, 400°), and with decrease in the pressure to 0.5 mmHg, the homogeneous reaction is not observed up to 500°.

The processes of deposition of compounds AB by the reactions of OMC with hydrides have been classified[85] on the basis of the stability of the deposition process and the possibilities for the production of defect-free stoichiometric layers. The $P-T-X$ diagrams for $A^{II}B^{VI}$ compounds were used to construct a hierarchy of pressures: $P_A^{AB(B)}$—the pressure of the vapour of A over a composition with an excess of B, $P_A^{AB(A)}$—the pressure above a composition with an excess of A, and P_A^{cr}—the critical pressure for the formation of nuclei of the substance A on a layer AB with an excess of A. The pressures for the component B, $P_B^{AB(B)}$, P_B^{AB}, and $P_B^{AB(A)}$ are defined similarly. The effective pressures of the atoms P^e are defined in terms of the fluxes of the atoms into the adsorption layer as follows:

$$k_1 P_{OMC} - k_{-1} P_X = (2\pi MkT)^{-1/2} P^e$$

where M is the mass of the OMC molecule or the hydride, and the other quantities were defined above in the examination of the decomposition of the OMC. The pressure P^e and hence the composition of the layer determined by it is measured from the pressure above a layer AB with the stoichiometric composition.

The situations arising during deposition are shown in Table 3. At the temperature T_1 the hydride and the OMC are stable, and the compound AB is deposited by reaction (1); above the temperature T_1, the hydride or OMC decompose. If the sum $P_A^e + P_A^{AB}$ is less than $P_A^{AB(A)}$, the compound AB with a composition in its range of existence will be deposited, but the density of vacancies of the element B increases. If $P_A^e + P_A^{AB}$ is greater than $P_A^{AB(A)}$, a two-dimensional liquid of the element A may be produced on the surface, and if $P_A^e + P_A^{AB(A)} > P_A^{cr}$, condensation of the element A begins. With increase in temperature above T_2, the same regular features will also be observed for the element B, but with allowance for the reaction of the element B with the element A.

Table 3. Relationship between the effective pressures and the composition of AB and the stability of the process.[81]

Temperature	Effective pressures	Composition	Mechanism	Stability
$T<T_1$	$P^e=0$	AB	VC	stable
$T_2>T>T_1$	$P_A^e + P_A^{AB} < P_A^{AB(A)}$	AB, B vacancies	VC	stable
	$P_A^e + P_A^{AB} > P_A^{AB(A)}$	AB(A)	VLS	unstable
	$P_A^e + P_A^{AB} > P_A^{кр}$	*	VLS	stable
$T>T_2$	$P_A^e + P_A^{AB} < P_A^{AB(A)}$	AB	VC	stable
	$P_B^e + P_B^{AB} < P_B^{AB(B)}$	AB	VC	stable
	$P_A^e + P_B^{AB} < P_A^{AB(A)}$	AB(A)	VC + VLS**	unstable
$T>T_2$	$P_B^e + P_A^{AB} > P_B^{AB(B)}$	AB (B)	VC + VLS	unstable

*Formation of inclusions of the element A in the layer is possible.

**Reaction between the atoms of the elements A and B takes place on the surface of the layer.

It should be noted that the growth conditions are unstable when the pressures P_A^{cr} or $P_A^{AB(A)}$ reach the boundary of formation of the two-dimensional condensate.[58] The mechanism of the production of the instability is as follows: on two-dimensional condensation, a liquid phase is produced on the surface; on its surface there is a sharp increase in the coefficients of condensation and reactive adhesion, leading to exhaustion of the starting materials and a change from the VLS to the VC mechanism; subsequently, accumulation of atoms takes place again with a change to the VLS growth mechanism, and the process is repeated. The factor responsible is the hysteresis associated with the condensation–volatilisation process on going from adsorption to the formation of nuclei of the liquid phase or two-dimensional condensates. Fig.2 gives the transverse cross-section of a zinc selenide specimen deposited from the vapour of the OMC and the hydride. In spite of the fact that the growth rate changed by a factor of two along the plane of the specimen (concentration wedge), the frequency of the fluctuations in the composition was constant. This proves that the fluctuations in the selenium concentration in the layer are due to a collective process involving the entire surface. The fluctuations were suppressed by decreasing the pressure in the reactor or by increasing the hydrogen selenide pressure. With decrease in pressure, stoichiometric layers were obtained, and with increase in the hydrogen selenide pressure, layers with an excess of selenium were produced.[85]

In recent years, greatest progress has been made in the preparation of layers of zinc selenide. This is a broad-zone material with lattice parameter close to that of gallium arsenide. ZnSe layers are obtained by the reaction of DMZ or DEZ with hydrogen selenide or dimethylselenium vapour in a stream of a carrier gas or in a vacuum at pressures of 0.1–5 mmHg. Stutius[64,182–186] obtained ZnSe layers by the reaction of the OMC and the hydride at a temperature of 300–350 °C at a reduced pressure of H_2 in

a vertical reactor with a support at an angle of 45° to the axis of flow. He obtained highly perfect layers of zinc selenide on supports of gallium arsenide and germanium, which gave a clearly defined edge band in the photoluminescence spectrum and a ratio $I_e/I_i > 10^3$ (I_i and I_e are the intensities of the impurity and edge photoluminescence).[182] Later papers[64,182] gave the photoluminescence spectra recorded at a lower density of the exciting radiation; the ratio I_e/I_i for films with a thickness of 4 μm was greater than 50, and that for layers with a thickness of 0.1 μm was 0.5. Layers of ZnSe alloyed with aluminium, with a specific resistance reaching 0.05 Ω.cm, have been obtained. The alloys were produced by introducing TMA vapour into the reagent flux.[183,184]

To obtain better matching of the lattice parameters of gallium arsenide and zinc selenide, ZnS_xSe_{1-x} layers with $x = 0.05$ are obtained by adding H_2S to the stream of H_2Se. The decrease in the stresses at the ZnS_xSe_{1-x}–GaAs boundary leads to an increase in the photoluminescence yield of the layer.[183] The dependence of x on the mole fraction of hydrogen sulphide in the stream of hydrogen selenide y was obtained, and an equation relating these parameters was given:

$$x = \frac{0.0159y}{1 - y(1 - 0.0159)} .$$

With change in x in the range 0–0.1, the intensity of the edge band remains constant, and the intensity of the impurity band decreases and has a minimum at $x = 0.05$. This indicates that the intensity of the photoluminescence band at 600 nm is related to the density of defects at the ZnSe–GaAs boundary. To obtain ZnSe (p-type) layers, experiments were carried out to alloy zinc selenide with nitrogen, phosphorus, and arsenic from ammonia, phosphine, and arsine vapour. Nitrogen gives finer levels in ZnSe, but conducting specimens could not be obtained.[64,184]

The deposition of ZnSe from OMC vapour and the hydride at atmospheric pressure of hydrogen has been studied. The dependence of the growth rate on the consumption of the OMC and the hydride, and on temperature, was obtained in the range 450–700°.[187,188] Up to 560°, the value of W is independent of temperature, but above this value it decreases with increase in temperature. With increase in the hydrogen selenide flux, the rate of growth of the layer decreased, and with change in the DEZ flux a maximum in the deposition rate was observed; at a ratio of the DEZ flux to the H_2Se flux greater than 4, growth took place from an adsorption layer enriched in zinc. At a temperature of 520°, inclusions with the composition $Ga_2Se_3.9ZnSe$ were formed at the support boundary.

Zinc selenide has been obtained by the reaction of DEZ with dimethylselenium.[189] The growth rate in the temperature range 350–750° is limited by the transport of the OMC to the support, and the dependence of W on the consumption of the OMC is similar to that discussed above.[187] The preparation of epitaxial layers of ZnSe from a $ZnMe_2$–$SeEt_2$–H_2Se–H_2 mixture (molar ratio 0.45 : 9 : 2.9 : 675) has been reported. Mirror-smooth ZnSe layers with a low level of stresses were obtained.[190] Epitaxial layers of ZnS, ZnSe, and ZnO have been obtained by the reactions of DMZ with the corresponding heterocyclic derivatives of the group VI elements. Single-crystal layers of ZnS and ZnSe were obtained on supports of GaAs,[100] and ZnO layers were obtained on sapphire supports.[191] The chief advantage of the use of heterocyclic derivatives is that it is possible to mix them beforehand with the DMZ vapour.

The possibility of using deposition from OMC vapour to prepare a n-ZnSe–p-GaAs hetero-junction of high quality, suitable for producing electroluminescent and opto-electronic instruments, has been studied.[192–195] Epitaxial layers of ZnSe were obtained by the reaction of DEZ with hydrogen selenide, carried out in a vacuum at temperatures of 300–450° and pressures of 0.1–5 mmHg. Alloys were produced by introducing TMA vapour into the reagent flux. Plates of AGP-1 and AGChTs grade gallium arsenide with a concentration of p-type carriers of 10^{19} cm^{-3} were used as supports. This gave highly perfect ZnSe layers with a structureless edge photoluminescence band and a mirror-smooth surface, with a ratio $I_e/I_i > 10^3$ for a layer thickness of 0.5–5 μm.[188,196] Induced emission was observed on these layers.[197] The resonator was obtained by splitting along the cleavage planes GaAs plates with a deposited ZnSe layer with a thickness of 6 μm, without preliminary treatment of the surface. The threshold density of optical pumping was 50 W cm^{-2} at 77 K. Lasing was also achieved at room temperature.

The specific resistance of the ZnSe (n-type) layers varied in the range 3×10^{-3}–1 Ω.cm with change in the concentration of n-type carriers in the range 2×10^{18}–10^{16} cm^{-3}. For the production of devices, it is necessary to obtain a perfect n-ZnSe–p-GaAs hetero-junction with a low resistance. Examination of the reactions at the ZnSe–GaAs boundary showed that the interaction of gaseous hydrogen selenide with the p-GaAs surface can lead to displacement of the p–n transition in gallium arsenide, the formation of an insulating layer at the boundary, and the production of structures of the type n–p–n–p; this has been confirmed experimentally.[193]

The intermediate layer at the ZnSe–GaAs boundary has been studied by photoluminescence methods. The spectra recorded at right angles to the surface of the specimen contained an impurity photoluminescence peak at 600 nm. Observation of the photoluminescence from the splitting showed that the emission at 600 nm is localised in a range of 0.1–0.2 μm adjoining the gallium arsenide surface. Since the exciting radiation is completely absorbed in zinc selenide at a thickness of less than 0.1 μm, the observation of the band at 600 nm in the photoluminescence spectrum indicates that it is excited as a result of ambipolar diffusion of the carriers. Comparison of the intensity of the impurity photoluminescence band from sections of the ZnSe layer with different thicknesses made it possible to measure the diffusion length in zinc selenide specimens, which amounted to 2.5 μm at 300 K and more than 6 μm at 77 K.[192] The formation of the intermediate layer is related to the catalytic decomposition of hydrogen selenide on the gallium arsenide surface. Thus when gaseous H_2Se is supplied to the reactor, drops of selenium decorate the GaAs surface at 300°.[196] In the deposition of ZnSe on GaAs, two sets of conditions are realised in succession; the first corresponds to hetero-epitaxy, with the catalytic decomposition of hydrogen selenide and the growth of a ZnSe layer with an excess of selenium, and the second corresponds to auto-epitaxy, with the growth of stoichiometric zinc selenide.

In Ref.198, deposition from OMC vapour and the hydride in a vacuum gave zinc selenide layers with a thickness of 0.03–0.1 μm, whose photoluminescence spectrum, in contrast to Ref.64, gave a ratio $I_e/I_i \cong 10$, indicating the absence of an intermediate layer at the ZnSe–GaAs boundary. The photoelectric properties of hetero-junctions have been studied for ZnSe specimens alloyed with aluminium to a level of 10^{16}–3×10^{-18} cm^{-3}. The carrier collection coefficient increased with increase in the concentration of carriers; relative to the number of photons reaching the

p–n junction, it amounted to 0.92–1.00 in the range 460–890 nm, and at wavelengths less than 460 nm it varied in the range 0.2–0.3; this indicates the long length of ambipolar diffusion in the epitaxial layers (Fig.5).

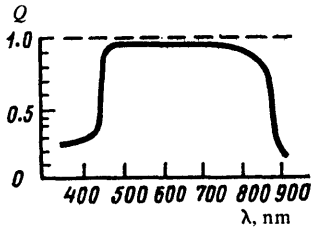


Figure 5. Dependence of the quantum efficiency Q of the hetero-junction p-GaAs–n-ZnSe on the wavelength of light.[198]

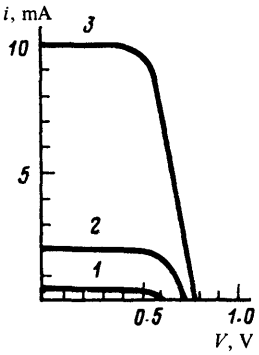


Figure 6. Load characteristics of a diode based on ZnSe–GaAs for different solar radiation powers: 0.4 MW cm^{-2} (*1*), 180 MW cm^{-2} (*2*), and 900 MW cm^{-2} (*3*).[198]

The forward and reverse branches of the voltage-current characteristic are approximated by the expressions:

$$i = i_0 \exp\left(\frac{V}{1.7\,kT}\right), \quad i = i_1 \exp\left[-C(V_0 - V)^{-\frac{1}{2}}\right]$$

where $i_0 = 10^{-8}$–10^{-9} A cm^{-2}, $C = 13$–14, and $V_0 = 1.30$–1.35 V. These forms of the forward and reverse branches of the voltage-current characteristic indicate that the predominant mechanism of current flow in the given diode is by tunneling.[199] The recording of the C–V characteristics of the hetero-junction showed that close to the ZnSe–GaAs interface there exists a layer of thickness 50 nm, which has a low concentration of carriers and which becomes narrower with increase in the concentration of carriers. The load characteristics of the diode were recorded using specimens with dimensions of 3 × 2 mm^2 without a brightening coating under direct solar radiation AM2 (Fig.6). The diode current was limited by the resistance of the contact, and not by the resistance of spreading; the efficiency of the diode varied in the range 15–18%, depending on the power of the incident radiation; the efficiency calculated with allowance for the production of a brightening coating was 18–22%.[198]

Experiments have been carried out to obtain zinc selenide from OMC vapour and the hydride at low carrier gas pressures. Conducting layers of zinc selenide were obtained without alloying at a deposition temperature of 250–300°; the conductivity decreased with increase in the deposition temperature to 370°, indicating uncontrolled alloying of the layers at low deposition temperatures.[200–202]

A number of studies have been devoted to the preparation of mercury telluride and the ternary system $Hg_xCd_{1-x}Te$.[203–206] The main difficulty in the production of this system is the high vapour pressure of mercury over compositions rich in mercury, which restricts the upper temperature of deposition to 400–420° and requires rapid cooling of the support with the epitaxial layer obtained when the deposition is complete. The deposition is carried out using a mixture of Hg–$CdMe_2$–$TeMe_2$–H_2 vapour, transported by a stream of hydrogen into the deposition zone. CdTe plates oriented with the (100), (110), or (111) faces are used as supports; the deposition temperature is 350–410°. An intermediate layer with a thickness of 0.4 μm, due to the diffusion of mercury in the support, was formed at the film–CdTe boundary. Increase in x was accompanied by a sharp change in the morphology of the surface of the layers; at $x < 0.2$, the surface was formed of irregular steps with cracks of a depth of up to 10 μm; at $x > 0.2$, the surface became mirror-smooth. Layers with an excess of tellurium showed n-type conductivity, and those with an excess of mercury showed p-type conductivity. The conductivity of the layers was determined by the consumption of mercury vapour in the reactor; the rate of deposition increased with increase in temperature.

A relatively small number of studies have been devoted to the preparation of epitaxial layers of $A^{IV}B^{VI}$ compounds. The deposition is carried out using the reaction of alkyls of group IV elements with hydrogen sulphide, hydrogen selenide, or tellurium alkyls in a stream of carrier gas (hydrogen). The temperature of deposition of the epitaxial layers was 550° for SnS, 500° for SnSe, 625° for SnTe, 550° for PbS and PbSe, 500° for PbTe, and 550–625° for $Pb_{1-x}Sn_xTe$.[52]

Table 4. Organobimetallic compounds used to prepare $A^{IV}B^{VI}$ compounds.[70–72]

Compound	T, °C	Solid products	Gaseous products
$(RS)_4Si$	500—600	SiS_2	SR_2
$(CH_3S)_2Cd$	400	CdS	SMe_2
$(R_3Ge)_2Hg$	400	Ge	C_1—C_4 (gas)
$Et_3SiTeGeEt_3$	450	GeTe	$TeEt_2$, EtH
$(Et_3Ge)_2Te$	450	GeTe	$GeEt_4$, $TeEt_2$
$(Et_3Ge)_2S$	450	GeS	$GeEt_4$
$Et_3GeTeEt$	450	GeTe	$GeEt_4$, $TeEt_2$
$(Et_3Sn)_2S$	350	SnS	$SnEt_4$
$(Et_3Sn)_2Te$	300	SnTe	$SnEt_4$, $SnEt_3H$
$Et_3SnTeEt$	400	SnTe	$TeEt_2$, $SnEt_4$

At lower temperatures, the compounds $A^{IV}B^{VI}$ can be obtained by the thermal decomposition of organobimetallic compounds containing a metal–chalcogen bond;[70–72] the deposition of the binary compound is identical to the deposition of the simple compound, for example of silicon or germanium. The process is simplified considerably, since the stage involving the mixing of the components is eliminated, making it possible to stabilise the stoichiometric

composition of the layer over the entire area of the support. Data on the preparation of epitaxial layers of compounds $A^{IV}B^{VI}$ from the vapour of organobimetallic compounds are given in Table 4. It can be seen from Table 4 that the deposition temperature of the layers is lower than that when a mixture of OMC vapour and the hydride is used.

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Thus deposition from OMC vapour is widely used to produce micro- and opto-electronic devices with exceptional parameters. New methods for the deposition of epitaxial layers are being rapidly developed and introduced into practice, the range of OMC used in technology is being expanded, and the search for new compounds is being continued. In addition, the theory of the deposition of epitaxial layers of binary compounds is being slowly developed (for 150–200 technical papers there is fewer than one paper on the study of the mechanism of the process), and this is holding up the introduction of methods for the deposition of epitaxial layers from OMC vapour in industry.

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