

Thermodynamic analysis of the deposition of GaAs epitaxial layers prepared by the MOCVD method

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On the basis of a detailed thermodynamic analysis of the Ga–As–C–H system, the initial conditions have been determined, under which the reaction of trimethylgallium (TMGa) and arsine in a hydrogen atmosphere produces a single condensed phase – solid GaAs. Liquid gallium with a small amount of dissolved arsenic is formed simultaneously when the initial ratio of the elements is $B^V/A^{III} < 1$, whereas solid graphite is simultaneously deposited at a high initial concentration of TMGa, especially at an elevated temperature and a decreased pressure. The equilibrium concentrations of the gaseous substances are strongly influenced by the initial B^V/A^{III} ratio. As_2 , As_4 , and CH_4 are the dominant species if $B^V/A^{III} > 1$, while CH_4 , $GaCH_3$, GaH and GaH_2 are the most abundant if $B^V/A^{III} < 1$. The calculated deposition diagrams are in good qualitative agreement with experimental results published in the literature. A comparison of the calculated composition of the gaseous phase and the results of experiments under the conditions used for the deposition of GaAs epitaxial layers leads to the conclusion that the course and results of the deposition process are significantly affected by transport and kinetic phenomena.

1. Introduction

Metallorganic chemical vapour deposition (MOCVD) is at present the most common technique used for the preparation of epitaxial layers and structures of the $A^{III}B^V$ type semiconductors to be applied in electronics and optoelectronics, especially on industrial production level [1–3]. In order to attain reproducible growth and high quality of the layers (uniform thickness and a required composition), all the factors affecting the course and the result of the deposition process must be studied in detail. The MOCVD method produces a layer of a required composition by a heterogeneous chemical reaction of initial gaseous substances (alkyl derivatives of A^{III} elements and hy-

drides or alkyl derivatives of B^V elements are commonly used) on the surface of a suitable substrate. The course and the result of the deposition process are thus simultaneously influenced by thermodynamic, kinetic and transport factors. Great attention has been paid in the literature to the thermodynamic aspects of the MOCVD process [4–13]. Although the diffusion of the initial substances towards the substrate or the surface chemical reaction are usually considered as the controlling processes, equilibrium calculations can provide valuable information, especially on the composition of the solid phase formed and its dependence on the deposition conditions.

Several authors have published the results of thermodynamic analyses of the Ga–As–C–H sys-

tem and calculated the homogeneous chemical equilibrium in the gaseous phase [10], as well as the heterogeneous chemical equilibrium [8,13], always including only a single condensed phase – solid GaAs – in the calculations. However, other condensed phases may also be formed, depending on the initial conditions: liquid gallium with a small amount of dissolved arsenic – (Ga–As) melt, solid graphite and solid arsenic. The present paper deals with a detailed thermodynamic analysis of the Ga–As–C–H system, with emphasis on the determination of the conditions under which a single condensed phase, solid GaAs, is thermodynamically stable in the system.

2. The calculation method, description of the system and the input thermodynamic data

The chemical equilibrium in the above system was calculated using a general method based on minimization of the total Gibbs energy of the system on a set of points satisfying the material balance conditions [14]. This is a modification of the White–Johnson–Dantzig method; The calcu-

lation program employs the algorithm described earlier by Eriksson [15].

Ideal behaviour of the gaseous phase was assumed in all the calculations and the following gaseous substances were included in the calculations procedure: Ga, Ga(CH₃)₃, Ga(CH₃), GaH₃, GaH₂, GaH, As₂, As₃, As₄, AsH₃, CH₄, C₂H₆, C₂H₄ and H₂. These substances were selected on the basis of our preliminary calculations and the results published in the literature [10,12,13]. Three condensed phases were further taken into the calculation, GaAs(s), the (Ga–As) melt and solid carbon. Solid arsenic was not considered, as the arsenic vapour pressure above As(s) is always higher within the studied temperature range [16] than that of the arsenic formed by dissociation of AsH₃ in a hydrogen atmosphere.

The input thermodynamic data for the individual substances were the standard molar Gibbs energies, $G_i^\circ(T)$ that are listed in table 1 for all the compounds. These values for the gaseous substances were calculated from the standard molar heats of formation $\Delta H_f^\circ(298\text{ K})$, the standard molar entropies, $S^\circ(298\text{ K})$ and the temperature dependences of the molar heat capacities, $C_p = f(T)$, which were from a single source [10] for all

Table 1
Input thermodynamic data: standard molar Gibbs energies G_i° (in kcal/mol)

Substance	$-G_i^\circ$ (kcal/mol)				
	$T = 600^\circ\text{C}$	$T = 650^\circ\text{C}$	$T = 700^\circ\text{C}$	$T = 750^\circ\text{C}$	$T = 800^\circ\text{C}$
Ga(g)	-113.324	-103.385	-93.387	-83.335	-73.231
Ga(CH ₃)(g)	170.773	187.117	203.680	220.453	237.431
Ga(CH ₃) ₃ (g)	401.813	427.977	454.702	481.974	509.775
GaH(g)	-36.456	-24.909	-13.270	-1.544	10.265
GaH ₂ (g)	46.330	59.874	73.551	87.357	101.286
GaH ₃ (g)	93.485	106.125	118.835	131.604	144.422
As ₂ (g)	33.732	47.704	61.778	75.946	90.206
As ₃ (g)	31.317	50.182	69.216	88.409	107.753
As ₄ (g)	154.476	174.610	194.968	215.538	236.310
AsH ₃ (g)	144.900	158.800	172.867	187.096	201.483
CH ₄ (g)	255.407	267.929	280.713	293.757	307.061
C ₂ H ₆ (g)	312.557	328.877	345.567	362.624	380.042
C ₂ H ₄ (g)	158.111	172.040	186.113	200.315	214.637
H ₂ (g)	124.884	133.108	141.426	149.834	158.331
Ga(l)	56.045	60.496	65.019	69.613	74.270
As(l)	35.771	40.078	44.468	48.937	53.476
GaAs(s)	157.879	163.912	170.084	176.389	182.816
C(s)	9.774	10.887	12.057	13.281	14.559

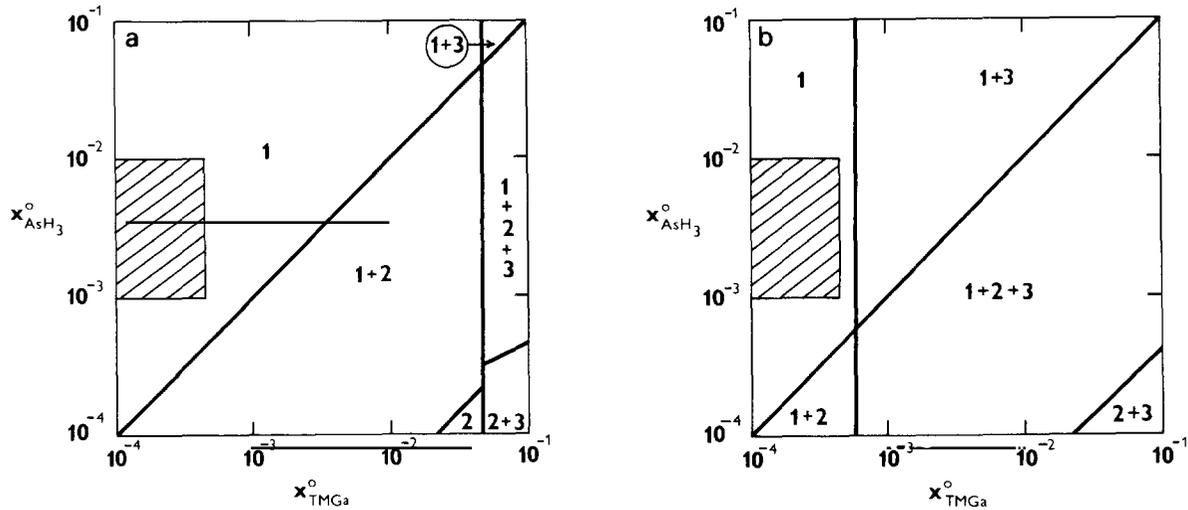


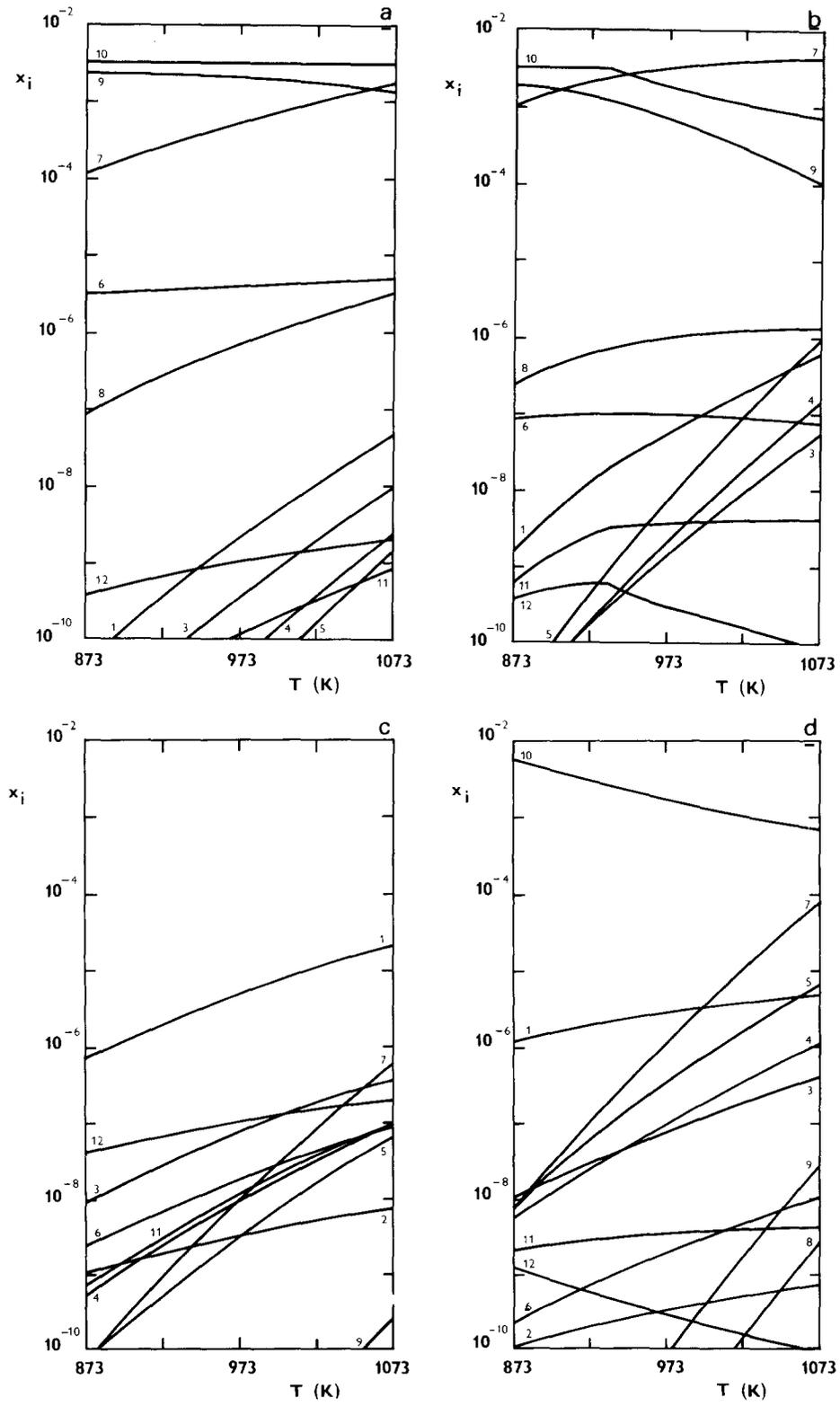
Fig. 1. The deposition diagram for the Ga-As-C-H system at a temperature of 700 °C and pressures of (a) 1 atm and (b) 0.01 atm (the marked region of the initial concentrations of TMGa and AsH₃ is usually used for the deposition of GaAs epitaxial layers): (1) GaAs(s); (2) (Ga-As)(l); (3) C(s).

Table 2

The calculated equilibrium amounts and compositions of the condensed phases in the Ga-As-C-H system for selected sets of initial conditions

<i>p</i> (atm)	<i>T</i> (°C)	<i>n</i> _{TMGa} ^o (mol)	<i>n</i> _{AsH₃} ^o (mol)	<i>n</i> _{Ga-As(l)} (mol)	<i>x</i> _{As(l)}	<i>n</i> _{GaAs(s)} (mol)	<i>n</i> _{C(s)} (mol)
1	600	1E-1	1E-3	9.9E-2	7.9E-4	9.2E-4	0
1	600	1E-1	1E-2	9.0E-2	7.9E-4	9.9E-3	0
1	600	1E-1	1E-1	0	0	1.0E-1	0
1	800	1E-1	1E-3	1.0E-1	9.9E-3	0	0.22
1	800	1E-1	1E-2	9.3E-2	1.7E-2	8.4E-3	0.22
1	800	1E-1	1E-1	0	0	1.0E-1	0.22
0.01	600	1E-1	1E-3	9.9E-2	7.9E-4	9.2E-4	0.29
0.01	600	1E-1	1E-2	9.0E-2	7.9E-4	9.9E-3	0.29
0.01	600	1E-1	1E-1	0	0	1.0E-1	0.29
0.01	800	1E-1	1E-3	1.0E-1	9.4E-3	0	0.30
0.01	800	1E-1	1E-2	9.3E-2	1.7E-2	8.2E-3	0.30
0.01	800	1E-1	1E-1	1.9E-3	1.7E-2	1.0E-1	0.30
1	600	1E-4	1E-4	0	0	1.0E-4	0
1	600	1E-4	1E-3	0	0	1.0E-4	0
1	600	1E-4	1E-2	0	0	1.0E-4	0
1	800	1E-4	1E-4	7.4E-7	1.7E-2	9.8E-5	0
1	800	1E-4	1E-3	0	0	1.0E-4	0
1	800	1E-4	1E-2	0	0	1.0E-4	0
0.01	600	1E-4	1E-4	0	0	1.0E-4	0
0.01	600	1E-4	1E-3	0	0	1.0E-4	0
0.01	600	1E-4	1E-2	0	0	1.0E-4	0
0.01	800	1E-4	1E-4	9.1E-5	1.4E-2	0	0
0.01	800	1E-4	1E-3	0	0	9.6E-5	0
0.01	800	1E-4	1E-2	0	0	9.9E-5	0

Note: 1E-4 = 1 × 10⁻⁴, etc.



substances involved, to ensure internal consistency. The values for solid GaAs were calculated from $\Delta H_f^\circ(\text{GaAs, s, 298 K})$ and $S^\circ(\text{GaAs, s, 298 K})$ published by Tmar et al. [17] and from $C_p = f(T)$ temperature dependence given in the tables of Barin et al. [18]. The data for Ga(l), As(s) and C(s) were also taken from these tables. To calculate the standard molar Gibbs energy of liquid As, the temperature dependence of the molar Gibbs energy of fusion for pure arsenic published by Gokcen [19] was further used.

The simple solution model [20] was utilized for the description of the thermodynamic properties of the (Ga–As) liquid phase. The activity coefficients γ_i for the components of a binary solution are then given by

$$RT \ln \gamma_i = \Omega_{ij}(1 - X_i)^2, \quad (1)$$

where Ω_{ij} is the interaction parameter in the i – j binary system that is independent of the solution composition but may, in general, be a function of the temperature. The value, $\Omega_{\text{Ga-As}} = 7782 - 25.1T$ (J/mol) was used and it was obtained from an optimization analysis of the published experimental data on the melt–solid phase equilibrium in the Ga–As system in a temperature range of 600 to 800 °C [21–25].

3. The calculation results

The chemical equilibrium in the Ga–As–C–H system was calculated for a temperature range of 600 to 800 °C (at 50 °C steps), at pressures of 1, 0.1 and 0.01 atm for various initial compositions

of the gaseous phase. The initial substances considered involved trimethylgallium (TMGa) ($n_{\text{TMGa}}^\circ = 10^{-1}, 10^{-2}, 10^{-3}$ and 10^{-4} mol), arsine ($n_{\text{AsH}_3}^\circ = 10^{-1}, 10^{-2}, 10^{-3}$ and 10^{-4} mol) and hydrogen ($n_{\text{H}_2}^\circ = 1 - n_{\text{TMGa}}^\circ - n_{\text{AsH}_3}^\circ$ mol in all the cases). The deposition diagrams for the Ga–As–C–h system at a temperature of 700 °C and pressure of 1 and 0.01 atm are shown in fig. 1. The calculated amounts and compositions of the condensed phase deposited are given in table 2 for selected sets of initial conditions. Fig. 2 depicts the equilibrium composition of the gaseous phase and its dependence on the temperature for various pressures and initial compositions of the system.

The following conclusions can be drawn from the calculated results:

(a) under common deposition conditions (i.e., $x_{\text{TMGa}}^\circ = 10^{-4}$ to 10^{-3} mol and the initial ratio $B^V/A^{\text{III}} > 1$), virtually all the gallium is transferred from the gaseous phase to solid GaAs, which is the only thermodynamically stable condensed phase in the system in the studied range of temperatures and pressures. The amount of the GaAs deposited slightly decreased with increasing temperature and decreasing pressure. Solid GaAs is also formed at an initial ratio of $B^V/A^{\text{III}} < 1$, when the liquid phase (Ga–As) is simultaneously formed. The decrease in the amount of the GaAs deposited with increasing temperature is then greater, as GaAs dissolves in the liquid phase until the melt is saturated with arsenic. The calculated equilibrium concentration of arsenic is $X_{\text{As}} = 7.870 \times 10^{-4}$ (600 °C), 4.353×10^{-3} (700 °C) and 1.704×10^{-2} (800 °C). GaAs is not formed at very low values of initial ratio B^V/A^{III} and at higher temperatures. The amount of the liquid phase deposited depends both on the TMGa initial concentration and the initial ratio B^V/A^{III} . At a given initial composition, the equilibrium amount of the liquid phase formed increases with increasing temperature and decreasing pressure.

(b) Solid graphite is simultaneously deposited at a high initial concentration of TMGa. The amount of carbon formed is relatively large due to the low thermodynamic stability of the carbon-containing gaseous substances and it increases with increasing temperature and decreasing pressure. Change in the initial B^V/A^{III} ratio only negligibly affects

Fig. 2. Dependence of the equilibrium composition of the gaseous phase in the Ga–As–C–H system on temperature (only those substances are given whose mole ratio is greater than 10^{-10}): (1) GaCH₃; (2) GaH₃; (3) GaH₂; (4) GaH; (5) Ga; (6) AsH₃; (7) As₂; (8) As₃; (9) As₄; (10) CH₄; (11) C₂H₄; (12) C₂H₆.

- (a) $n_{\text{TMGa}}^\circ = 10^{-3}$ mol, $n_{\text{AsH}_3}^\circ = 10^{12}$ mol, $p = 1$ atm;
 (b) $n_{\text{TMGa}}^\circ = 10^{-3}$ mol, $n_{\text{AsH}_3}^\circ = 20^{-2}$ mol, $p = 0.01$ atm;
 (c) $n_{\text{TMGa}}^\circ = 10^{-2}$ mol, $n_{\text{AsH}_3}^\circ = 10^{-3}$ mol, $p = 1$ atm;
 (d) $n_{\text{TMGa}}^\circ = 10^{-2}$ mol, $n_{\text{AsH}_3}^\circ = 10^{-3}$ mol, $p = 0.01$ atm.

the amount of the carbon formed at a constant initial concentration of TMGa.

(c) The relative contents of the substances in the gaseous phase are strongly influenced by the initial conditions. Arsenic occurs in the gaseous phase primarily in the form of As_2 and As_4 ; As_2 being more stable at a decreased pressure and higher temperature and As_4 being more stable at atmospheric pressure and a lower temperature. The equilibrium concentrations of As_3 and AsH_3 are several orders of magnitude lower. The elemental Ga and its compounds are highly unstable in the gaseous phase, within the given ranges of temperature and pressure. Relatively the most stable compounds appear to be the GaCH_3 radical, than the hydrides GaH and GaH_2 and, under a decreased pressure, also Ga. Carbon is mainly bound in CH_4 in the gaseous phase. In the case of a low initial concentration of TMGa, the amount of methane is virtually independent of the temperature at a pressure of 1 atm and roughly equals three times the initial amount of TMGa. Under a decreased pressure and an elevated temperature, the equilibrium amount of CH_4 decreased due to the formation of solid carbon. The other hydrocarbons and organometallic compounds and radicals are present in negligible amounts.

4. Discussion

The calculated equilibrium concentrations of the gaseous substances, their relative stabilities and the dependencies of the equilibrium composition of the gaseous phase on temperature, pressure and the initial composition of the system under the conditions when only solid GaAs is stable among all the condensed phases considered are in agreement with the results of Chadwick [13]. It is difficult to compare the calculated values with experimental data, because the composition of the gaseous phase in the Ga–As–C–H system has not been studied under equilibrium conditions, but usually under the conditions employed for deposition of GaAs epitaxial layers. Complete decomposition of the initial TMGa has been demonstrated by IR [26–28] and mass [29] spectroscopy and CH_4 , has been detected as a gaseous product.

The amount of the methane formed is virtually constant and corresponds to about three times the initial amount of TMGa, which is in agreement with the calculation results. Gaseous substances containing gallium atoms have not been directly determined. The results of Haigh and O'Brien [30], who followed the contents of gaseous gallium in the products of the thermal decomposition of TMGa using atomic absorption spectroscopy, were unable to demonstrate the presence of gallium, which indicates that gallium is highly unstable and thus present at a very low concentration in the gaseous phase; this has also been predicted from our equilibrium calculations.

Several authors investigated pyrolysis of AsH_3 in the $\text{AsH}_3 + \text{H}_2$ and $\text{AsH}_3 + \text{TMGa} + \text{H}_2$ systems, Ban [31] indicated As_2 and As_4 as the main products of decomposition of AsH_3 in a hydrogen atmosphere, at atmospheric pressure and a temperature of 850°C , using mass spectroscopy; the degree of conversion of AsH_3 was 0.99 under these conditions. The thermal decomposition of AsH_3 was further studied by IR spectroscopy [26–28]. Under atmospheric pressure, the degree of conversion of AsH_3 in the $\text{AsH}_3 + \text{H}_2$ was 0.5 for a temperature of 660 to 680°C (cf. ref. [26]) and 530 to 560°C (cf. ref. [27]). The presence of TMGa leads to an increase in the AsH_3 conversion and a temperature of 490 to 500°C suffices for attaining of a degree of conversion of 0.5 [27]. The CARS method (coherent anti-Stokes Raman scattering) was used by Lückerrath et al. [32] for “in situ” analyses of the AsH_3 pyrolysis and it was found that a temperature of 150 to 200°C was sufficient for a degree of conversion of 0.5; at temperatures above 600°C , the AsH_3 decomposition is almost complete. These authors monitored hydrogen as a product of AsH_3 pyrolysis in the $\text{AsH}_3 + \text{N}_2$ system and found out that the amount of hydrogen liberated, especially at lower temperatures, was less than the total amount of hydrogen from decomposed AsH_3 and thus the pyrolysis products also involved the hydrides AsH_2 and AsH . Monteil et al. [33] arrived at the same conclusion on the basis of the broadening of the vibrational bands in the AsH_3 Raman spectrum on partial decomposition of the substance. All the above experimental results thus indicate a greater

stability of gaseous arsine than that predicted by equilibrium calculations and suggest the presence of the hydrides AsH_2 and AsH that, on the basis of equilibrium calculations, are present among the products of the AsH_3 thermal decomposition under given conditions only in negligible concentrations. This finding and the fact that the decomposition of arsine is significantly affected by the presence of solid GaAs through a catalytic surface action [27,28] leads to the conclusion that the course of the deposition process is significantly influenced by the kinetics of the homogeneous and heterogeneous chemical reactions that may occur in the Ga-As-C-H system.

It is possible to compare some of the calculated phase boundaries of the deposition diagrams with experimental results. Several authors [34–37] have pointed out that surfaces showing defects have been grown when conditions are outside an optimum range of reactant flow. It has been suggested that for low gas phase As concentration, liquid Ga droplets form on the surface, leading to a pseudo-VLS mechanism formation of hillocks. The gallium droplet formation has been recently demonstrated by Larsen et al. [38]. They have deposited GaAs layers at initial $\text{B}^{\text{V}}/\text{A}^{\text{III}}$ ratios of 2.0 and 0.5, and the resulting surfaces were examined under a microscope. In the first case the surfaces were smooth and specular, but when TMGa was in excess, Ga droplets sitting atop GaAs whiskers were observed.

The calculations suggest that solid carbon is not deposited under commonly used deposition conditions. However, the presence of carbon in GaAs layers has been demonstrated experimentally [39–41]. This is probably caused by the fact that carbon is not present in the system as a separate phase, but is dissolved in solid GaAs, although to a very limited extent. The work on refinement of the thermodynamic model for contamination of GaAs layers with carbon is in progress.

5. Conclusion

The conditions under which solid GaAs is formed as the only condensed phase in the Ga-

As-C-H system have been determined on the basis of a detailed thermodynamic analysis of this system. These conditions, especially the initial composition of the gaseous phase, are in agreement with those commonly used for deposition of GaAs epitaxial layers by the MOCVD method which ensures a good morphology of the layers produced. It follows from the calculated equilibrium composition of the gaseous phase that the most stable gaseous substance containing gallium is GaCH_3 , those containing arsenic are As_4 and As_2 and that with carbon CH_4 . A comparison of the calculated composition of the gaseous phase and the results of experiments under the conditions used for deposition of GaAs layers leads to the conclusion that the course and result of deposition process are significantly affected by kinetic processes, homogeneous ones in the gaseous phase and heterogeneous ones at the interface of the gaseous phase and solid GaAs. Still, thermodynamic analysis is an important part of physico-chemical treatment of the deposition process and our further work is devoted to a refinement of the thermodynamic model of contamination of GaAs layers by carbon.

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