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Kinetics and mechanism of thermal decomposition of GaN

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Abstract

A scheme of dissociative evaporation of GaN with the partial evolution of nitrogen in the form of free atoms has been invoked to interpret the decomposition mechanism of GaN in vacuum or inert gas atmosphere. A critical analysis of literature data and their comparison with theoretical calculations has shown that the main kinetic characteristics of decomposition, including the absolute decomposition rate and activation energy are in full agreement with the reaction: $GaN(s) \rightarrow Ga(g)+0.5N+0.25N_2$. Condensation of the gallium vapor in the reaction zone and partial transport of condensation energy to GaN account for the features which are typical of many solid-state reactions and manifest themselves in the appearance of induction and acceleration periods in the course of the process. The low temperature decomposition of GaN in H_2 according to the equilibrium reaction $GaN(s)+1.5H_2=Ga(g)+NH_3$ is supported by a good agreement of experimental and calculated activation energies and by the strong inhibition effect of NH_3 on the GaN decomposition. As expected, condensation of Ga vapor in the Ga(l)/GaN(s) interface accelerates the reduction of GaN by H_2 several hundred times.

Keywords: Activation energy; Autocatalysis; Dissociative evaporation; Gallium nitride; Kinetics; Mechanism

1. Introduction

Gallium nitride is a semiconductor with a large direct band gap which makes this material interesting in short-wavelength photonic devices for display and storage applications. Its high thermal conductivity also opens new routes in high-temperature/high-power electronics. Knowledge of GaN decomposition conditions and mechanism is vital for film growth, processing of devices and high-temperature operation.

There are not many studies in the literature concerning GaN decomposition though the first report was published by Johnson et al. more than 60 years ago [1]. In 1964, Searcy [2] estimated a coefficient for congruent vaporization of GaN of the order 10^{-6}. In 1965, Munir and Searcy [3] measured by a torsion-effusion method the heat of activation for the reaction $2GaN(s) \rightarrow 2Ga(l)+N_2$ and by a torsion-Langmuir method the rate and heat of activation for the reaction $2GaN(s) \rightarrow 2Ga(g)+N_2$. In 1965, Schoonmaker et al. [4] determined the pressure over GaN and GaN+Ga mixture by weight loss effusion and torsion effusion techniques. The results supported that the vaporization coefficient for the decomposition is much less than unity. In contrast to that, the binary mixture (a direct contact of GaN with liquid Ga) showed considerable dissociation. The authors [4] proposed that a non-reactant liquid Ga may participate as catalysts in the vaporization process by dissolving GaN and disrupting its rigid wurtzite crystal structure.

The decomposition of GaN in the presence of H_2 was first studied by Thurmond and Logan [5,6] in the 1970s. Products of the reaction are liquid gallium and

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ammonia gas. A comparison of the NH_3 to H_2 partial pressure ratio at the exit of the reactor revealed a very good agreement with thermodynamic calculations which means that this reaction is close to equilibrium. Jacob et al. [7] also measured the decomposition of GaN in different gases and found a value of initial decomposition temperature 970°C in Ar/N_2 and 600°C in H_2.

Ambacher et al. [8] have measured the flux of N_2 and activation energy for GaN decomposition in vacuum by a quadropole mass spectrometer (QMS). The enhanced GaN decomposition rate in H_2 in the presence of liquid gallium has been supported recently by Koleske at al. [9] and Pisch and Schmid-Fetzer [10]. Tanaka and Nakadaira [11] and Rebey et al. [12] have investigated the GaN decomposition at various temperatures in H_2 and measured the activation energy of this process.

The objective of this work is to discuss the mechanism and the above-mentioned features of GaN decomposition in vacuum, H_2 and in the presence of liquid gallium using a scheme of dissociative evaporation of the reactant with simultaneous condensation of the low-volatile product. This approach has been employed earlier to explain the mechanism and kinetics of the thermal decomposition of nitrates [13–15], azides [16], carbonates [17], $Li_2SO_4\cdot H_20$ [18], $Mg(OH)_2$ [19], Ag_2O [20], HgO [21], and of a number of other inorganic compounds [22–24], including GaN. In the last case, the analysis was based on the experimental results of only one work [3] and it was concluded that the GaN decomposition in vacuum occurs with the evolution of 40% of nitrogen in the form of free atoms, i.e. by the reaction $GaN(s) \rightarrow Ga(g)+0.4N+0.3N_2$.

2. Theoretical

The method to be employed below consists in comparing experimental data for the kinetic parameters with their theoretical values. The calculations are based on the classical evaporation model of Hertz–Langmuir, extended to the cases of dissociative evaporation of compounds. The scheme for the theoretical calculation of the main kinetic parameters (the flux of the gaseous product J, the rate constant k, the product partial pressure P and the parameters of the Arrhenius equation, E_a and A) has been described in a number of previous publications [13–22]. Therefore, we are going to present below only some final relations necessary for the calculations in this work.

In the case of a binary compound S decomposed in vacuum into gaseous products A and B

$$\mathrm{S(s)} \rightarrow a\mathrm{A(g)} + b\mathrm{B(g)} \tag{1}$$

the flux of product A can be expressed through the equivalent partial pressure P_A (in atm) of this product corresponding to the hypothetical equilibrium of reaction (1) in the form

$$J_A = \frac{\gamma M P_A}{(2\pi M_A R T)^{1/2}} \tag{2}$$

where M and M_A are the molar masses of the reactant and product A, respectively, γ the coefficient of conversion from atmospheres to Pascals, and R is the gas constant.

A theoretical value of the partial pressure of product A can be calculated from the equilibrium constant K_p for reaction (1). In the absence of an excess of reaction products in the reactor atmosphere, the situation corresponding to the *equimolar* evaporation mode, the partial pressure P_A can be expressed [23] as

$$P_A^e = a\left(\frac{K_P}{F}\right)^{1/v}\left(\frac{M_A}{M_B}\right)^{b/2v} = \frac{a}{F^{1/v}}\left(\frac{M_A}{M_B}\right)^{b/2v}\exp\left(\frac{\Delta_r S_T^0}{vR}\right)\exp\left(-\frac{\Delta_r H_T^0}{vRT}\right) \tag{3}$$

where

$$F \equiv a^a \times b^b \tag{4}$$

$$v = a + b \tag{5}$$

and

$$K_P = P_A^a \cdot P_B^b \tag{6}$$

Here $\Delta_r H_T^0$ and $\Delta_r S_T^0$ are, respectively, the changes of the enthalpy and entropy in process (1).

As can be seen from Eq. (3), the calculated activation energy for reaction (1) can be written as

$$E_a^e = \frac{\Delta_r H_T^0}{v} \tag{7}$$

In order to take into account the partial transfer of the energy released in the condensation of low-volatile A product to the reactant, we introduced, as before [18–21,24], into calculations of the enthalpy of

Table 1
Thermodynamic functions [27–29] used in the calculations

Species	State of aggregation	ΔH^0_{1300} (kJ mol^{-1})	S^0_{1300} (J mol^{-1} K^{-1})
Ga	l	37.8	98.7
Ga	g	302.4	206.3
GaN	s	−58.9[a]	120.7[a]
N_2	g	40.2	236.8
N	g	497.8	183.8
H_2	g	38.4	174.2
NH_3	g	21.5	261.9

[a] The entropy and heat capacity of ZnO (an isoelectronic solid) were used in the evaluation of these functions.

decomposition reaction (1) an additional term $\tau a \Delta_c H^0_T(\mathrm{A})$, where the coefficient τ corresponds to the fraction of the condensation energy transferred to the reactant. Thus, we can write

$$\Delta_r H^0_T = a\Delta H^0_T(\mathrm{A}) + b\Delta H^0_T(\mathrm{B}) - \Delta H^0_T(\mathrm{S}) + \tau a \Delta_c H^0_T(\mathrm{A}) \quad (8)$$

The most plausible of all conceivable mechanisms of the energy transfer appears to be the thermal accommodation [25,26] or, in other words, direct transfer of the energy at the reaction interface in collisions of the low-volatile molecules with the reactant and product surface. For equal temperatures of the solid phases, one may expect equipartition of energy between the two phases, i.e. τ=0.5. Table 1 lists the values of the thermodynamic functions [27–29] for all components of the assumed reactions.

3. Results and discussion

3.1. Decomposition of GaN in vacuum

In accordance with our previous interpretation of the low coefficient of GaN decomposition [22], we attribute the anomalous thermal stability of GaN to the specific features of its wurtzite crystalline structure with very strong covalent bonds between each nitrogen atom and its four nearest gallium neighbors and, as a result of this, to the partial evolution of nitrogen in the process of thermal decomposition in the form of free atoms. Actually, the distance of closest approach of two nitrogen atoms in a lattice of GaN is about 3.2 Å, whereas the N–N internuclear distance in the gaseous N_2 molecule is only 1.07 Å. There are no structural units in the condensed phase which are similar to gaseous N_2 molecule. Therefore, it is difficult to expect that all nitrogen atoms of the solid GaN evolve in the process of its decomposition in the form of N_2 molecules. By analogy with our analysis of the mechanism of decomposition of azides [16], we can represent the decomposition reaction as

$$\mathrm{GaN(s)} \rightarrow \mathrm{Ga(g)} + (1-i)\mathrm{N} + 0.5i\mathrm{N}_2 \quad (9)$$

where the interaction parameter i varies from 0 to 1, depending on the extent to which the nearest nitrogen atoms interact with one another in the instant of decomposition.

To select the appropriate value of interaction parameter (or a part of nitrogen evolved in a molecular form), we used a comparison of experimental and theoretically calculated kinetic parameters, namely, the activation energies and absolute rates of decomposition. Table 2 presents the results of this comparison. Some comments to these data should be made. In the case of E_a value obtained in [3], we used a correction for the self-cooling effect at high temperatures. The method of correction has been described elsewhere [19]. Ambacher et al. [8] used two different techniques for the determination of decomposition rate. The first one was based on direct measuring of the N_2 flux from epitaxial GaN by mass spectrometry in the temperature range from 1125 to 1270 K. The

Table 2
Activation energy and partial pressure of nitrogen (reduced to N_2) for decomposition of GaN in vacuum at τ=0

ΔT (K)	E_a (kJ mol^{-1})		$\overline{T}$ (K)	P (atm)	
	Experimental	Calculated		Experimental	Calculated
1166–1428	313[a] [3]	354	1300	9.4×10^{-8} [3]	5.0×10^{-8}
1125–1270	379 [8]	354	1200	2.6×10^{-9} [8]	3.2×10^{-9}
1123–1223	288 [8]	354	1173	2.0×10^{-9} [8]	1.4×10^{-9}

[a] After correction of the original value (305 kJ mol^{-1} [3]) for the self-cooling effect.

second technique (used to verify the data obtained by the first technique) consisted of measuring the GaN film thickness by X-ray diffraction and determination of the amount of GaN which was removed by thermally induced decomposition at temperatures of 1123, 1173 and 1223 K.

The best agreement as a whole between the experimental and calculated values corresponds to i=0.5 (50% of evolved nitrogen is in the form of N_2 and 50%, in the form of free atoms) or the reaction

$$\mathrm{GaN(s)} \rightarrow \mathrm{Ga(g)}+0.5\mathrm{N} + 0.25\mathrm{N_2} \qquad (10)$$

Under this supposition, the discrepancy between the equivalent partial pressures of nitrogen corresponded to the fluxes of nitrogen at the mean temperatures of experiments and equilibrium partial pressures for the reaction implied is within a factor of two. The values of activation energy compared are in a good agreement as well, if we take into account that the lesser slope of Arrhenius plot (E_a=288 kJ mol^{-1} [8]) is determined by only three points and, therefore, is the least reliable.

There are some other facts which support the suggested scheme of GaN decomposition. Firstly, it is supported by the absence of a strong depressive effect of nitrogen atmosphere on the decomposition of GaN. As reported in several works [10,12,30,31], the difference in the decomposition rate of GaN in Ar (or He) and N_2 is rather small (in the range of factor of 2). Note that in case of evolution of nitrogen in the molecular form it would be higher of one order of magnitude. Secondly, GaN cannot be prepared via a direct syntheses of Ga(l/g) and N_2 (under not very high pressures). At the same time, it can be prepared by reacting Ga with atomic N generated from N_2 by microwave discharge [32] or radio-frequency fields [33]. The last argument in favor of the evolution of atomic nitrogen is a strong adsorption of nitrogen (about 90%) at the reactor walls observed by Ambacher et al. [8] in their vacuum experiments on the decomposition of GaN. It may be suggested that free N atoms evolved are chemisorbed on the surface of reactor (quartz) or metal surface of QMS.

In connection with these mass spectrometry experiments, a remark can be made. The most reliable and obvious argument for, or against, the decomposition mechanism could be obtained in a mass spectrometric investigation of the primary products of decomposition. This technique was used by L'vov and Novichikhin [14,15] in investigations of the decomposition mechanism of some metal nitrates. Later on, the same approach was used by L'vov [16] for the interpretation of decomposition mechanism of azides. In the latter case, the mass spectrometric data obtained by Walker and co-workers [34–36] were used. The primary condition for the right application of this technique, which is necessary for preventing possible adsorption, condensation or recombination of unstable species evolved from a sample surface, is the elimination of any collisions of these species with furnace and spectrometer walls on their way to the QMS ionizer. In the experimental setup used by Ambacher et al. [8], the furnace was set perpendicular to ionizer. Therefore, any direct tracks of species from the sample to the QMS were excluded. This explains the absence of any appreciable m-s signals in the measurement of GaN decomposition [8] not only for atomic nitrogen (as one of the primary decomposition species) but even for gallium atoms.

Let us turn now to the explanation of the catalytic effect of liquid gallium on the decomposition of GaN. The origin of these effect has been interpreted recently in the framework of the mechanism of congruent dissociative evaporation with simultaneous condensation of the low-volatility product at the reactant/product interface [20,24,37]. The essence of this approach is the transfer of one-half of the condensation energy to the reactant and, as a result, increasing of its decomposition rate. In the case of the decomposition of GaN, it means that the enthalpy of reaction (10) is reduced from 629.3 (τ=0) to 488.0 kJ mol^{-1} (τ=0.5) and the equilibrium partial pressure of Ga at 1300 K increased from 9.9×10^{-8} up to 1.1×10^{-4} atm. The saturated vapor pressure of Ga at 1300 K is 9.7×10^{-6} atm. Therefore, in the presence (or after formation) of GaN(s)/Ga(l) interface, the decomposition of GaN should occur with the simultaneous condensation of Ga vapor and the initial temperature of GaN decomposition should be reduced from 1300 to 1100 K.

The experimental data are in agreement with these theoretical estimations. Munir and Searcy [3] observed in their effusion experiments the appearance of drops of elemental gallium in the Knudsen cell with 1.4 mm orifice, when the ratio of orifice area to upper surface of sample was about 1/100 and the partial pressure of Ga at 1300 K reached ca. 3×10^{-5} atm, i.e. three-fold of its saturated pressure. For the Knudsen cell with 3 mm orifice and about 1/30 ratio of orifice area to upper surface of sample, the partial pressure of Ga corresponded to ca. 1×10^{-5} atm and no elemental gallium remained in the cell.

A further proof of the proposed mechanism are the results of investigation by Schoonmaker et al. [4] of the catalytic effect of liquid gallium on GaN decomposition by weight loss and torsion effusion techniques. In agreement with the previous work [3], they observed a black residue of finely divided gallium covered the surface of the initially pale yellow nitride in all runs (with different effusion cells), except those with very large orifices (2.3 mm diameter). The next comment is remarkable: "As expected, the effusion rate increased with increasing of temperature. However, another curious result was observed. In successive runs at the same temperature with the same sample of initially pure, crystalline gallium nitride the rate of effusion, calculated from weight loss, increased from one run to the next". In the framework of our theory, the reason of this effect is rather obvious. It is connected with increasing the degree of formation of GaN(s)/Ga(l) reaction interface. This increasing process should continue up to the moment when the gallium film covers the whole surface of GaN and the decomposition reaction reaches a steady state.

Another important result of this work has been the demonstration of GaN decomposition catalysis by the liquid Ga metal added to GaN samples. No decomposition of pure, crystalline GaN occurred after 180 min of heating at 1189 K in the open container. After addition of a ball of liquid gallium into container, the sample of GaN began to decompose and the decomposition pressure slowly increased at constant temperature (1189 K). The accelerating period (up to the maximum equivalent pressure 1.4×10^{-5} atm) was about 50 min. In case of better contact between Ga metal and GaN, the induction period is considerably reduced. The above mentioned value of equivalent pressure coincides with the calculated equilibrium pressure for reaction (10) at the steady state condition $\tau=0.5$. Therefore, in all instances (in the presence as well as in the absence of Ga metal), the decomposition of GaN in vacuum occurs in agreement with the proposed scheme.

Lastly, Schoonmaker et al. [4] have shown that the decomposition of GaN is catalyzed not only by Ga, but In metal as well. This fact is also in agreement with the theoretical model proposed and the experimental observations of the catalytic effect of foreign impurities on the decomposition of other solids, for example, of metallic Ni on the decomposition of Ag_2O [38].

3.2. Decomposition of GaN in hydrogen

Most of the researchers explain the low temperature decomposition of GaN in H_2 by the reaction

$$GaN(s) + 1.5H_2 = Ga(g) + NH_3 \tag{11}$$

which is the reverse reaction to that used for a synthesis of GaN. This is supported by a good agreement of experimental and calculated activation energies for this reaction (Table 3). There are some other facts which confirm the equilibrium of this reaction. In the experiments by Rebey et al. [12], at the initial decomposition temperature 600°C (873 K) and 1 atm H_2, the equilibrium pressure of Ga vapor should be about 3×10^{-8} atm. An addition to H_2 a small amount ofNH_3 (3%) inhibited the decomposition of GaN [12]. Indeed, in this case (at 873 K) the equilibrium pressure of Ga was reduced to 4×10^{-14} atm. Only at 1250 K, its value should reach the same 3×10^{-8} atm (as in the absence of NH_3 at 873 K). The same inhibition effect of NH_3 on the decomposition of GaN has been observed by Furtado and Jacob [39].

The high partial pressure of Ga vapor in the decomposition of GaN in H_2 results in its condensation and formation of droplets of liquid metal on the surface of sample. Koleske et al. [9] studied this process at different pressures of H_2 (10–700 Torr) and two different temperatures: 1174 and 1265 K. For pressures less than 40 Torr no Ga droplets were observed. This is in agreement with the theoretical calculations. At 1265 K and 40 Torr H_2, the equilibrium partial pressure of Ga for reaction (11) is smaller than its saturated

Table 3
Activation energy for the decomposition of GaN in H_2

Conditions	ΔT (K)	E_a (kJ mol^{-1})	
		Experimental	Calculated
7 l min^{-1} H_2 flow, 0.1 atm	1085–1186	155 [11]	163
3 l min^{-1} H_2 flow, 1 atm	931–1070	180 [12]	163

pressure (3.8×10^{-6} and 4.9×10^{-6} atm, respectively). At 76 Torr of H_2, when Ga droplets were observed, the equilibrium partial pressure of Ga (6.1×10^{-6} atm) is 24% greater than the saturated vapor pressure. The remarkable accord between the results of these equilibrium calculations and observations supports the validity of thermodynamic functions for GaN presented in Table 1.

The presence of liquid gallium catalyses the reaction (11) in the same way as it does in the vacuum decomposition of GaN. It explains 'a dramatic increase in the GaN decomposition rate correlated with the increased Ga surface accumulation' observed by Koleske et al. [9]. The maximum rate of GaN decomposition in H_2 and in the presence of liquid gallium should correspond to $\tau=0.5$ condition. At 1265 K and 76 Torr H_2, as an example, the equilibrium partial pressure of Ga in reaction (11) should be increased to 3.3×10^{-3} atm, which is several hundred times higher than that in the absence of liquid gallium (6.1×10^{-6} atm). The equivalent partial pressure of Ga that corresponded to the decomposition of GaN at these conditions (1265 K and 76 Torr H_2) was equal to 1.3×10^{-4} atm [9]. This is 25 times lower than expected but it should be taken into account that the degree of covering of GaN surface by liquid Ga in this experiment was very small.

4. Conclusions

As it follows from the above discussion, all the peculiarities of thermal decomposition of GaN in vacuum or in an atmosphere of hydrogen can be quantitatively interpreted and predicted in the framework of the scheme of dissociative evaporation of the reactant with simultaneous condensation of the low-volatile product. The reason for the high thermal stability of GaN in vacuum or in inert gases is in its wurtzite crystalline structure with strong covalent bonds between each nitrogen atom and its four nearest gallium neighbors and the long distances between nitrogen atoms in the lattice. As a result of these, the partial evolution of nitrogen in the process of thermal decomposition is in the form of free atoms. In relation to equilibrium

$$2\mathrm{GaN(s)} = 2\mathrm{Ga(l)} + \mathrm{N_2} \qquad (12)$$

the vaporization coefficient for decomposition of GaN in vacuum is as low as 1×10^{-8} at 1300 K and 4×10^{-10} at 1000 K. It can be expected that this feature (the partial evolution of diatomic gases in the form of free atoms) will be observed not only for Al and In nitrides, but for some other O–, S– or Se–containing compounds with a wurtzite structure. Our preliminary estimations [22] support this conclusion.

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