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# SOME THERMODYNAMIC ASPECTS OF NITRIDES IN MATERIALS SCIENCE
## Fluorine bomb calorimetry study

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The energies of combustion in fluorine of gallium nitride and indium nitride in wurzite crystalline structure have been measured in a two-compartment calorimetric bomb, and new standard molar enthalpies of formation have been calculated: $\Delta_f H_m^0$(GaN(cr) 298.15 K)=–(163.7±4.2) kJ mol$^{-1}$ and $\Delta_f H_m^0$(InN(cr) 298.15 K)=–(146.5±4.6) kJ mol$^{-1}$. Comparison with the recommended values of the $\Delta_f H_m^0$ nitrides from the literature is also presented.

***Keywords:*** *calorimetry, fluorine combustion, gallium nitride, indium nitride, standard molar enthalpy of formation*

## Introduction

Many inorganic nitrides (from IV and III group periodic table) were classified recently as the 'high-tech' materials. It was estimated, because of theirs unusual thermal stability, resistance to the oxidation, and the excellent physical properties at the high temperatures. The results for the standard molar enthalpies of formation $\Delta_f H_m^0$ of some nitrides from IV and III groups obtained in the past by fluorine combustion calorimetry were introduced in the book [1] and next papers [2–4]. The fluorine bomb calorimetry, a descendant of the classical oxygen bomb calorimetry, has been used in studies of inorganic substances that cannot be made to combine readily or completely with oxygen. Previously the fluorine-combustion calorimetric studies of the some nitrides from IV and III group ($Si_3N_4$ and BN) were performed at the National Institute of Standards and Technology (NIST). The other nitrides from III group (Ga, In, Al) are very important as semiconductors [5]; therefore, it is necessary to control their thermodynamic properties. For example, detailed knowledge of the GaN and InN thermodynamic properties would be helpful in optimizing its crystal growth. However the collected data standard molar enthalpy of formation for $\Delta_f H_m^0$(GaN, 298.15 K) found in the papers have vary all the way from –111.2 [6] to –263.6 kJ mol$^{-1}$ [7], and the results for $\Delta_f H_m^0$(InN, 298.15 K) are from –19.2 [6] to –143.5 kJ mol$^{-1}$ [8]. We were making plan to attain the thermodynamic properties for the technologically important III group nitrides and to test the standard molar enthalpies of formation $\Delta_f H_m^0$. Recently a new laboratory for fluorine-combustion calorimetry has been set up at the Institute of Physical Chemistry (IPC). To check up results the standard molar enthalpy of formation of a gallium nitride $\Delta_f H_m^0$(GaN, 298.15 K) and of an indium nitride $\Delta_f H_m^0$(InN, 298.15 K) we applied the new research by use the fluorine combustion calorimetry. The inorganic substances containing nitrogen ($Si_3N_4$, BN) have been before successfully studied by fluorine bomb calorimeter at NIST because they ignite on contact with high-pressure fluorine at room temperature and yield $N_2(g)$ along with well-defined fluorides in gaseous phase ($SiF_4(g)$, $BF_3(g)$). In our previous experiments it soon that crystalline specimens of GaN(cr) and InN(cr) did not ignite spontaneously in $F_2(g)$ at the experimental pressure so it was necessary to use a combustion aid conjunct with a fuse.

## Experimental

### *Materials*

Sample of monocrystalline GaN(cr) in hexagonal wurzite structure was received from High Pressure Research Center PAS (Warsaw, Poland). The GaN(cr) sample was prepared by high-pressure and high-temperature crystallization method described by Grzegory *et al*. [11]. According to X-ray diffraction analysis, the sample was pure and there was no evidence for the presence of additional compounds.

Sample of InN(cr) black powder of crystalline wurzite structure was manufactured by Sigma-Aldrich with analysis of In (89.3% by gravimetric assay) and trace metals. The X-ray diffraction pattern and thermal analysis experiments show that in sample indium nitride

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was the amount of pure In (8.4±3%). Sample was also analysed by Perkin-Elmer Elemental Analyser in Institute of Organic Chemistry PAS for the following elements (in mass%): N, 9.86; C, 0.22; H, 0; S, 0; Cl, 0.

The other products used in calorimetric experiments were: fluorine of 99% purity purchased from Messer Germany, which passed through the column filled with NaF to remove any trace of HF; high-purity rhombohedral sulfur (sample USBM-P1b from the NIST laboratory) [12] and high-purity hexagonal selenium (batch no. S75045) from Johnson Matthey [13] used as ignition aids. Also tungsten foil (of 99.99 mass% from Schwarzkopf Technologies Corp.) used here as an auxiliary combustion, was formed into saucers of diameter, as described previously [14].

### *Methods*

#### Instrument

Laboratory for fluorine-combustion calorimetry has been set up at IPC, and was made similar to the arrangement at NIST. Details of the construction and operation of the calorimetric instrument are described by O'Hare [9]. The calorimetric system contained an isoperibol calorimeter and two-compartment (bomb+tank) calorimetric vessel designed for compounds reacting with fluorine. The fluorine storage tank was charged ($T \approx 293$ K) to a pressure of 1 MPa for the experiments with samples. Calorimetric experiment started after the calorimetric vessel is immersed in a can, filled with an accurately mass amount of water and placed in calorimetric thermostat. The temperatures of the can and thermostat are monitored by probes of the quartz-crystal thermometer Hewlett-Packard and registered on PC computer.

Before the experiments with fluorine, the calorimetric system was calibrated by combustion in high-purity oxygen of benzoic acid with a specific energy of combustion certified to be $-26434 \pm 3$ J g$^{-1}$ under prescribed conditions. The energy equivalent was determined to be $\varepsilon_{calor}=(13926.0 \pm 1.1)$ J K$^{-1}$ (the uncertainty is the standard deviation of the mean). The bomb under prescribed conditions was preconditioned by initial exposure to $F_2$. To the calorimetric system contains also a manifold, placed in ventilated hood, used for transferring high-pressure fluorine to and from the bomb and gaseous fluorides from the combustion [10]. The UNI-LAB glovebox has been purchased from M. Braun Company (85748 Garching, Germany). This glovebox with a purification unit and circulating by the $N_2(g)$ is used to storing all the samples, which must be dry in order to obviate adsorption of $H_2O$ and $O_2$. Also all operations with samples prepared in the open bomb must be taken in the glovebox.

#### Calorimetric experiments

Exploratory experiments showed that our specimens of GaN(cr) and InN(cr) did not ignite spontaneously in $F_2(g)$ at the experimental pressure and their fluorides products of the reaction created in the solid phase. It was necessary to use a tungsten saucer as an auxiliary combustion, in conjunction with a fuse. With the GaN was applied as a fuse selenium reacting spontaneously with fluorine: $Se+3F_2(g)=SeF_6(g)$, and with InN was used a sulfur, also reacting spontaneously: $S+3F_2(g)=SF_6(g)$. Combustion with the fuse initiated the reaction of fluorine with the tungsten to $WF_6(g)$ and with a nitride. Gallium nitride would react with fluorine as follows:

$$GaN(cr)+3/2F_2(g)=GaF_3(cr)+0.5N_2(g) \qquad (1)$$

The reaction of indium nitride with fluorine was similar to reaction (1) of gallium nitride.

$$InN(cr)+3/2F_2(g)=InF_3(cr)+0.5N_2(g) \qquad (2)$$

Gallium and indium products of combustion (1, 2) were in the solid phase in the crucible and in the bomb, so they have to be analyzed by X-ray diffraction and in the measurements, they were pure. The energies of combustion in fluorine of crystalline GaN and InN have been measured in the two-compartment calorimetric bomb at 298.15 K.

## Results and discussion

### *Calorimetry of GaN*

Details of the calorimetric measurements on reaction (1) are given in Table 1. Many of the symbols used in this table have been defined by Hubbard [15]. Masses of materials placed in the bomb are denoted by $m$. Thus, $\Delta\theta_c$ is the corrected temperature increase; $\varepsilon_{calor}$ is the energy equivalent of the system; $\Delta U$(cont) is a correction of the net energy equivalent of the contents of the bomb, calculated from values of the heat capacities; $\Delta U$(gas) is a standard-state correction, that was calculated by use of the intermolecular-force constants from the literature for $F_2$ [16], $WF_6$ [17] and $N_2$ [18]; the small quantities of $SeF_6(g)$ have been neglected. The quantity $\Delta U$(blank), measured in separate experiments by expansion of $F_2(g)$ was calculated as outlined in O'Hare publication [19]. The quantities $\Delta U$(W) and $\Delta U$(Se) give the contributions to the total energy measured in the experiment. Corrections for the fluorination of the tungsten to the $WF_6$ and of the selenium to $SeF_6$ were derived from massic energies of combustion in $F_2$ of W [14] and with Se fuse [13]. They are denoted by $\Delta U(W)/J=-(9374.4 \pm 3.1)$ J g$^{-1}\cdot m^{-1}$(W)/g and $\Delta U(Se)/J=-(14097 \pm 6)$ J g$^{-1}\cdot m^{-1}$(Se)/g. The massic energy of combustion $\Delta_c u$ of GaN was calculated as the sum of

**Table 1** Massic energy of combustion $\Delta_c u$ of GaN(cr) in fluorine; $T$=298.15 K, $p^0$=101.325 kPa $\varepsilon_{calor}$=(13926.0±1.1) J K$^{-1}$

| | 1 | 2 | 3 | 4 | 5 | 6 |
|---|---|---|---|---|---|---|
| $m$(GaN)/g | 0.18957 | 0.12294 | 0.12205 | 0.12491 | 0.12400 | 0.10988 |
| $m$(W)/g | 0.77926 | 0.79109 | 0.80339 | 0.90556 | 0.70710 | 0.70677 |
| $m$(Se)/g | 0.05161 | 0.04925 | 0.04311 | 0.05606 | 0.04964 | 0.05357 |
| $\Delta\theta_c$/K | 0.74121 | 0.79109 | 0.80339 | 0.90556 | 0.70710 | 0.70677 |
| $\Delta U$(W) | 7304.2 | 7415.0 | 7530.3 | 8488.0 | 6627.8 | 6624.7 |
| $\Delta U$(Se) | 727.4 | 694.3 | 607.7 | 790.3 | 699.8 | 755.2 |
| $\Delta U$(cont) | –4.659 | –4.332 | –4.321 | –4.805 | –3.967 | –3.898 |
| $\Delta U$(blank) | 7.62 | 7.62 | 7.62 | 7.62 | 7.62 | 7.62 |
| $\Delta U$(gas) | 0.572 | 0.568 | 0.580 | 0.513 | 0.554 | 0.535 |
| $(-\Delta\theta_c)\varepsilon_{calor}$ | –10322.5 | –9586.4 | –9606.6 | –10787.1 | –8835.3 | –8709.8 |
| $\Delta_c u$/J g$^{-1}$ | –12065.9 | –11983.0 | –12000.3 | –12052.9 | –12125.3 | –12066.1 |

$^{a}\langle\Delta_c u\rangle = -(12048.8\pm19)$ J g$^{-1}$

$^{b}\Delta_c u^0 = -(12048.8\pm50)$ J g$^{-1}$

[a] Uncertainty is the standard deviation of the mean of the individual values of $\Delta_c u$

[b] The final uncertainty $2s=2(\Sigma s_i^2)^{1/2}$ comprises the accumulated individual uncertainties expressed as standard deviations.

**Table 2** Summary of values of the $\Delta_f H_m^0$(GaN(cr) 298.15 K)

| Investigators | Method | $\Delta_f H_m^0$/kJ mol$^{-1}$ |
|---|---|---|
| Madar [22] | estimated from P,T data | –152.1 |
| Wood *et al*. [7] | fluorine bomb calorimetry | –263.6 or –167.4 |
| Karpiński and Porowski [21] | estimated from P,T data | –157.8 |
| Hahn and Juza [6] | oxygen bomb calorimetry | –110±3.8 |
| this work | fluorine bomb calorimetry | –163.7±4.2 |

the energy contributions in each column divided by the corresponding mass of sample $m$(GaN). The mean value of energy of combustion were then derived from individual values with uncertainty determined as standard deviation of the mean $\langle\Delta_c u\rangle = -(12048.8\pm19)$ J g$^{-1}$. The standard massic energy of combustion of monocrystalline GaN is $\Delta_c u^0$(GaN)= $-(12048.8\pm50)$ J g$^{-1}$. On the basis of the molar mass $M_{GaN}$=83.73 g mol$^{-1}$ we derive $\Delta_c U^0 = -(1008.8\pm4.2)$ kJ mol$^{-1}$ and for reaction (1) the difference the numbers of moles of gaseous products and substrates is $\Delta\nu^g=0.5-1.5=-1$ and $\Delta\nu^g RT = -2.48$ kJ mol$^{-1}$. The molar enthalpy of combustion of reaction (1) was calculated: $\Delta_c H_m^0 = \Delta_c U^0 + \Delta\nu^g RT = -1011.3\pm 4.2$ kJ mol$^{-1}$.

According to reaction (1): $\Delta_c H_m^0 = \Delta_f H_m^0(GaF_3) - \Delta_f H_m^0$(GaN(cr)) combined with value of gallium trifluoride $\Delta_f H_m^0(GaF_3(cr)) = -1175$ kJ mol$^{-1}$ from Gurvich *et al*. [20] yield standard enthalpy of formation $\Delta_f H_m^0$(GaN(cr) 298.15 K)=–163.7 ±4.2 kJ mol$^{-1}$. The formation reaction at $T$=298.15 K and $p^0$=101.325 kPa is:

$$Ga(s)+0.5N_2=GaN(cr) \qquad (3)$$

Other results of $\Delta_f H_m^0$(GaN) taken from the literature are presented in the Table 2.

The value of the enthalpy of formation $\Delta_f H_m^0$(GaN(cr) 298.15 K)=–157.8 kJ mol$^{-1}$ reported by Karpiński *et al*. [21], calculated on the basis of the measured experimentally equilibrium pressure of $N_2$ over GaN, seems to be quite close to our result: –163.7±4.2 kJ mol$^{-1}$.

## *Calorimetry of InN*

Experimental results of the massic energy of combustion $\Delta_c u$ of InN are given in Table 3, where the symbols are the same as in Table 1. The quantity $\Delta U$(S) gives the contributions to the total energy measured in the experiment and correction for the fluorination of the rhombohedral sulfur fuse to $SF_6$ was derived from massic energy of combustion in $F_2$ with S(cr, rhomb) [14]. The data were calculated by $\Delta U$(S)/J=–(37956±13) J g$^{-1}\cdot m$(S)/g. The mean value was $\Delta_c u = -(8396.1\pm7.6)$ J g$^{-1}$, but to determine the standard massic energy it was necessary to calculate the corrections of impurities that existing in InN sample. Analytical results showed that in sample InN exists pure In, that reacts with $F_2$ making up the same

**Table 3** Massic energy of combustion $\Delta_c u$ of InN(cr) in fluorine; $T$=298.15 K, $p^0$=101.325 kPa, $\varepsilon_{calor}$=(13926.0±1.1) J K$^{-1}$

| | 1 | 2 | 3 | 4 | 5 |
|---|---|---|---|---|---|
| $m$(InN)/g | 0.13367 | 0.08199 | 0.07703 | 0.07623 | 0.12816 |
| $m$(W)/g | 0.52028 | 0.46021 | 0.47505 | 0.47761 | 0.39172 |
| $m$(S)/g | 0.01635 | 0.02473 | 0.00195 | 0.00202 | 0.00220 |
| $\Delta\theta_c$/K | 0.47546 | 0.42673 | 0.37152 | 0.37287 | 0.34689 |
| $\Delta U$(W) | 4876.7 | 4313.6 | 4452.7 | 4476.7 | 3671.7 |
| $\Delta U$(S) | 620.6 | 938.6 | 74.0 | 76.7 | 83.5 |
| $\Delta U$(cont) | –4.712 | –4.192 | –3.601 | –3.615 | –3.401 |
| $\Delta U$(blank) | 4.0 | 4.0 | 4.0 | 4.0 | 4.0 |
| $\Delta U$(gas) | 0.541 | 0.464 | 0.632 | 0.632 | 0.627 |
| $(-\Delta\theta_c)\varepsilon_{calor}$ | –6621.5 | –5942.9 | –5174.0 | –5192.8 | –4831.0 |
| $\Delta_c u$/J g$^{-1}$ | –8412.1 | –8419.7 | –8389.3 | –8374.5 | –8384.8 |

$^a\langle\Delta_c u\rangle = -(8396.1\pm7.6)$ J g$^{-1}$

Impurity correction=(317.1±19.7)

$^b\Delta_c u^0 = -(8079.7\pm36)$ J g$^{-1}$

[a] Uncertainty is the standard deviation of the mean of the individual values of $\Delta_c u$

[b] The final uncertainty $2s=2(\Sigma s_i^2)^{1/2}$ comprises the accumulated individual uncertainties expressed as standard deviations

**Table 4** Impurity corrections for InN(cr) sample (Sigma Aldrich, black powder); $w$ denotes mass fraction

| Impurity | $w\cdot10^2$ | Combustion products | $\Delta_c u$(imp)/kJ g$^{-1}$ | $\Delta(\Delta_c u)^a$/kJ g$^{-1}$ | $w\Delta(\Delta_c u)$/J g$^{-1}$ |
|---|---|---|---|---|---|
| In – pure | 8.4 | $InF_3$ | –10.331 | 1.935 | 162.5 |
| C – free carbon | 0.22 | $CF_4$ | –77.76 | 69.36 | 152.6 |
| Metals | | | | | 2.02 |

Impurity correction: (317.12±19.7) J g$^{-1}$

[a] $\Delta(\Delta_c u)=\Delta_c u(\text{InN})-\Delta_c u$ (imp); correction calculated on the basis of determined values determined value $\Delta_c u(\text{InN})=-(8396.1\pm7.6)$ J g$^{-1}$ and $\Delta_c u$(imp) values calculated using auxiliary data $\Delta_f H_m^0$ of formation $InF_3$ and $CF_4$

**Table 5** Corrections for trace metals impurities in InN(cr) sample (Sigma Aldrich)

| Impurity | $w\cdot10^6$ mass fraction | Assumed impurity form | $W\cdot10^6$ mass fraction impurity form | $\Delta_f H_m^0$(imp)$^a$/ kJ mol$^{-1}$ | $\Delta_c u$(imp)/ kJ g$^{-1}$ | $\Delta(\Delta_c u)^b$/ kJ g$^{-1}$ | $W\Delta(\Delta_c u)$/ J g$^{-1}$ |
|---|---|---|---|---|---|---|---|
| Na | 51.6 | Na | 51.6 | 0 | –24.97 | 16.57 | 0.85 |
| Ca | 27.0 | $Ca_3N_2$ | 30.14 | –430.99 | –21.87 | 13.47 | 0.41 |
| Fe | 21.8 | $Fe_4N$ | 23.17 | –11.09 | –11.81 | 3.41 | 0.079 |
| Ga | 17.3 | GaN | 20.77 | –163.7 | –12.05 | 3.65 | 0.076 |
| Te | 17.0 | SnTe, $Ag_2Te$ | | | | | |
| Sn | 8.4 | SnTe | 17.43 | –1955.6 | –9.03 | 0.639 | 0.011 |
| Ag | 5.9 | $Ag_2Te$ | 17.64 | –1742.2 | –7.90 | –0.496 | –0.009 |
| Mg | 5.9 | $Mg_3N_2$ | 8.17 | –92.0 | –71.88 | 63.48 | 0.52 |
| Cr | 4.7 | CrN | 5.97 | –75.31 | –17.44 | 9.04 | 0.05 |
| Ni | 1.7 | $Ni_3N_2$ | 1.97 | –311.71 | –21.39 | 12.99 | 0.026 |
| Zn | 1.7 | $Zn_3N_2$ | 1.94 | –22.6 | –9.59 | 1.19 | 0.002 |

Impurity correction: 2.02 J g$^{-1}$

[a] $\Delta_f H_m^0$(imp) – standard molar enthalpy of formation of the impurity. Their values and $\Delta_f H_m^0$ combustion products with $F_2$ were taken from Barin [23]. Combustion products are: NaF, $CaF_2$, $FeF_2$, $GaF_3$, $TeF_6$, $SnF_2$, AgF, $MgF_2$, $CrF_3$, $NiF_2$, $ZnF_2$ and $N_2$

[b] $\Delta_c u=\Delta_c u(\text{InN})-\Delta_c u(\text{imp})$; correction calculated on the basis of determined value $\Delta_c u(\text{InN})=-(8396.1\pm7.6)$ J g$^{-1}$ and $\Delta_c u$(imp) values calculated using auxiliary data $\Delta_f H_m^0$

**Table 6** Summary of values of the $\Delta_f H_m^0$(InN(cr), 298.15 K)

| Investigators | Method | $\Delta_f H_m^0$/kJ mol$^{-1}$ |
|---|---|---|
| Jones and Rose [24] | static | –131.4±6.7 |
| Gorodienko [27] | Knudsen | –127.6±2.5 |
| Vorob'ev *et al*. [25] | Knudsen | –136.4±18.8 |
| Vorob'ev *et al*. [26] | Knudsen | –132.6±6.3 |
| Hahn and Juza [6] | oxygen bomb calorimetry | –19.2±3.8 |
| McChesney *et al*. [8] | calorimetric | –143.5 |
| this work | fluorine bomb calorimetry | –146.5±4.6 |

product $InF_3$. It was assumed that impurity C was present as free carbon, and the trace metals are presented as nitrides and tellurides, only without Na (probably sodium azide $NaN_3$ can not exist in this sample).

The data impurity correction calculated in Tables 4 and 5 is: (317.1±19.7) J g$^{-1}$.

The standard massic energy of combustion is thus $\Delta_c u^0=-(8079.7\pm36)$ J g$^{-1}$. On the basis of the molar mass $M_{InN}$=128.8267 g mol$^{-1}$ was derived molar energy $\Delta_c U^0=-(1040.9\pm4.6)$ kJ mol$^{-1}$, and according the reaction (2) molar enthalpy of combustion is following: $\Delta_f H_m^0=\Delta_c U^0+\Delta v^g RT=-(1043.4\pm4.6)$ kJ mol$^{-1}$ where $\Delta v^g RT=-2.48$ kJ mol$^{-1}$.

According to reaction with fluorine (2): $\Delta_c H_m^0=\Delta_f H_m^0(InF_3)-\Delta_f H_m^0$(InN(cr)) combined with value of indium trifluoride $\Delta_f H_m^0(InF_3(s))=-1189.9$ kJ mol$^{-1}$ from [20] yield standard enthalpy of formation $\Delta_f H_m^0$(InN(cr) 298.15 K)=–(146.5±4.6) kJ mol$^{-1}$. The formation reaction of InN(cr) at $T$=298.15 K and $p^0$=101.325 kPa is following: $In(s)+0.5N_2=InN(cr)$. Our result of $\Delta_f H_m^0(InF_3(s)$ 298.15 K) is close with two values [8, 26] presented in Table 6.

Combustion of InN in oxygen bomb made by Hahn [6] was not effective.

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