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Compensation effect in the kinetics of chemical treatment of GaAs, InAs, GaSb, and InSb using bromine-releasing etching compositions based on H_2O_2–HBr solutions for the development of effective chemical polishing techniques

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In this work, the kinetic dependences of chemical-dynamic etching of semiconductor compounds GaAs, InAs, GaSb, and InSb in polishing etching mixtures based on H_2O_2–HBr without and with an organic solvent are investigated. Based on the temperature dependence of the polishing kinetics in these systems, the apparent activation energies and pre-exponential factors in the Arrhenius equation for the chemical dissolution of the crystal surfaces were calculated. The existence of a kinetic compensation effect between the activation energy and the pre-exponential factor during the heterogeneous surface polishing process of GaAs, InAs, GaSb, and InSb was established. It was shown that the compensation dependence is not affected by the presence of an organic solvent in the bromine-releasing etching composition or by the chemical nature of the $A^{III}B^{V}$-type semiconductor material. The introduction of an organic solvent into the H_2O_2–HBr etching mixture facilitates the transition of the InSb dissolution process from the kinetic to the diffusion-controlled regime.

Keywords: Arrhenius equation, apparent activation energy, kinetic compensation effect.

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Introduction

The use of semiconductor materials in the modern production of high-performance devices and electronic components remains highly relevant. However, the demands on the quality of processed single-crystal surfaces, their intrinsic properties, and the combination of these properties within the material are continually increasing, including the need for optimized synthesis methods of semiconductor materials that take into account predefined material characteristics and physicochemical parameters. One of the critical parameters is the quality of the treated surface of single crystals and epitaxial films in the active region of a device. To obtain high-quality polished crystal surfaces, liquid-phase etching methods are typically employed particularly chemical-mechanical polishing (CMP) and chemical-dynamic polishing (CDP).

GaAs, InAs, GaSb, and InSb are considered promising materials for the fabrication of lasers, integrated circuits, photodiodes, and photodetectors for the infrared spectral region, solar cells based on InGaAs/GaAsPN LED structures, field-effect transistors, heterojunction bipolar transistors, and vertical-cavity surface-emitting lasers. Indium-based compounds (InAs, InSb) are also widely used for the development of ultrafast devices with minimal energy consumption [1–10].

The CDP and CMP processes for semiconductor materials of GaAs, InAs, GaSb, and InSb surfaces belong to the class of heterogeneous activation processes occurring at the solid–liquid interface. One of the key factors influencing the kinetics of the chemical interaction between the liquid etching medium and the semiconductor surface is the temperature at which this interaction takes place. The rate constant of the chemical reaction depends on the activation energy of the process, which can be experimentally determined based on the temperature dependence of the reaction rate. The relationship between the reaction rate constant and the activation energy is described by the Arrhenius equation:

$$k = C_E e^{-Ea/RT} \tag{1}$$

where k – is the reaction rate constant, C_E – is the pre-exponential factor (collision frequency of particles), E_a – is the activation energy of the process, and R – is the universal gas constant *($R = 8.314\ J{\cdot}mol^{-1}{\cdot}K^{-1}$).*

Based on the temperature dependence of the chemical interaction kinetics, it is possible to determine the apparent activation energy and the value of the pre-exponential factor. An increase in the activation energy of the chemical polishing process of semiconductor surfaces implies a decrease in its rate, since fewer particles have sufficient energy to participate in the chemical interaction. The calculated apparent activation energy E_a reflects only the enthalpic component of the process. However, the overall course of the process is governed by the Gibbs free energy, which includes both enthalpic and entropic contributions:

$$\Delta G = \Delta H - T\Delta S \tag{2}$$

The entropic factor is influenced by the shape and size of the particles, their phase state, orientation at the moment of collision, as well as by phase transitions and diffusion processes – all of which affect the interaction rate. The change in entropy is associated with the pre-exponential factor according to the following relationship:

$$C_E = e^{\Delta S/R} \tag{3}$$

The effect of various chemical interaction factors in heterogeneous systems on enthalpic and entropic components, which overall do not significantly alter the Gibbs free energy, is known in the literature as the kinetic compensation effect [11]. The presence of this effect indicates similarity in the type of chemical reactions and a common mechanism [12]. Specifically, an increase in activation energy is accompanied by an increase in the entropy of the process, and conversely, a decrease in activation energy is accompanied by a reduction in system entropy, which ultimately does not lead to significant changes in the kinetics of chemical interaction in heterogeneous systems. Reactions are considered to be of the same type if they differ only by the structure of one reactant or by the experimental conditions, such as the use of different catalysts, media, etc.

I. Experimental Procedure

The study was carried out using single-crystal wafers of various crystallographic orientations of both undoped and doped GaAs, GaSb, InAs, InAs(Sn) (tin-doped), and InSb semiconductors:

GaAs – *n*-type, (100) orientation, undoped;

GaSb – semiconductor grade, unoriented, undoped;

InAs – $n = 5{\times}10^{16}$ cm^{-3}, $\mu = 3{\times}10^4$ cm^2/(V·s), (111) orientation, undoped;

InAs – *n*-type, (100) orientation, undoped;

InAs(Sn) – $n = 2.6{\times}10^{16}$ cm^{-3}, $\mu = 2.6{\times}10^4$cm^2/(V·s), (111) orientation, tin-doped;

InSb – *n*-type, (211) orientation, undoped.

Before the experiments, the surfaces of the single-crystal samples (with an area of 0.5–0.6 cm^2) were mechanically pre-polished using abrasive powders with progressively decreasing grain sizes (from ASM 7/5 to ASM 1/0). Before chemical etching, a mechanically damaged layer (100–150 μm) was removed from the working surface by pre-etching with a universal etchant for $A^{III}B^V$ compounds, composed (by vol.) of: 6 HNO_3 – 10 HF – 4 H_2SO_4, followed by treatment with the experimental etching solutions.

The regularities of chemical etching of GaAs, InAs, InAs(Sn), GaSb, and InSb in aqueous solutions of H_2O_2–HBr–solvent were studied using a setup for CDP, based on the rotating disk method [13], at a rotation speed of $\gamma = 86$ min^{-1} and a temperature range of T = 285–305 K. The etching solutions were prepared from a mixture of 40% HBr and 35% aqueous H_2O_2, with added aqueous solutions of tartaric, citric, or lactic acids to form complex ions with the reaction products and to regulate the oxidizer concentration and the viscosity of the etching mixture. The etching compositions were prepared immediately before CDP, following a specific order of mixing and allowed to stand for 2 h to reach chemical equilibrium and to dissolve the free bromine in the resulting mixture. Bromine evolution occurs as a result of the reaction between the initial components of the etching solution:

$$H_2O_2 + 2HBr = Br_2 \uparrow + 2H_2O \tag{4}$$

The dissolution rate was determined by measuring the change in thickness of the single-crystalline samples before and after etching, using a 1-MIGP dial indicator with a precision of ±0.5 μm. Two to five samples were etched simultaneously. The samples were fixed in a fluoroplastic holder and secured with a wide outer ring. This ensured all specimens were positioned in the same plane. Such configuration promoted uniform etching and minimized the occurrence of turbulent flow within the etching solution.

To determine the apparent activation energy (E_a), the temperature dependence of the semiconductor dissolution rate was plotted in coordinates of *ln v ~ 1/T*, and the slope of the resulting straight line was used to calculate E_a.

According to [14], E_a values for diffusion-limited processes generally do not exceed 25–30 kJ·mol^{-1}, while dissolution processes limited by the rate of chemical reactions can exhibit E_a values significantly higher than 30–35 kJ·mol^{-1}.

II. Experimental Results and Discussion

The pre-exponential factor in phase-boundary reactions may vary over a wide range. For activation processes of the same type that proceed via a common mechanism, a correlation is typically observed between the apparent activation energy (E_a) and the pre-exponential factor (C_E), this relationship has been noted for many classes of activated processes, including heterogeneous catalysis, gas desorption from solid surfaces, and catalytic decomposition of secondary alcohols [7].

The chemical polishing of semiconductor crystal surfaces is also an activated process, and thus, a linear relationship between $ln\,C_E$ and E_a is expected, which is described by the equation:

$$ln\,C_E = aE_a + b \qquad (5)$$

where a and b are constants [15].

This relationship is described in the literature for heterogeneous and catalytic processes and is referred to as the *kinetic compensation effect* [16]. This effect suggests that an increase in the entropic factor C_E in the Arrhenius equation fully or partially compensates for the exponential suppression due to $(-\,E_a/RT)$.

The chemical etching of GaAs, InAs, GaSb, and InSb in bromine-releasing solutions based on H_2O_2–HBr also represents an activated process, and the existence of a compensation effect is expected [16]. By plotting the kinetic etching data for GaAs, InAs, GaSb, and InSb in the investigated solutions using the function $ln\,v = f(1/T)$, the apparent activation energies (E_a) and pre-exponential factors C_E of the polishing process were calculated. Their values varied considerably, with E_a ranging from 2.0 to 51.2 kJ·mol^{-1}, and $ln\,C_E$ from 0.8 to 3.1 (see Table 1).

A linear correlation between $ln\,C_e$ and E_a was established by plotting $ln\,C_E = f(E)$, and all obtained values were found to lie on straight lines. This linear dependence indicates the presence of a *kinetic compensation effect.*

For the investigated semiconductor compounds, 26 pairs of values for the apparent activation energy and the pre-exponential factor were obtained (Table 1).

The obtained values indicate that the rate-limiting steps of the CDP of GaAs, InAs, GaSb, and InSb surfaces with bromine-releasing etching mixtures are diffusion-controlled, since the calculated apparent activation energy does not exceed 30 kJ·mol^{-1}; it is confirmed by the observed dependence of the reaction rate on the rotation speed of the disk. Graphical dependencies of $ln\,C_E$ versus E_a were constructed for the polishing process of the investigated semiconductor materials in bromine-releasing solutions. In the case of polishing the semiconductors GaAs, InAs, GaSb, and InSb with a solution containing (in vol.%) $6H_2O_2$ and 94HBr (Fig. 1), the dependence is described by the equation:

$$ln\,C_E = (0{,}033 \pm 0{,}001) \cdot E_a + (1{,}461 \pm 0{,}022) \qquad (6)$$

Table 1.

Apparent activation energy (***E_a***) and logarithm of the pre-exponential factor (ln ***C_E***) for the dissolution process of GaAs, InAs, GaSb, and InSb in H_2O_2–HBr–solvent solutions

Material	Solution composition (vol.%)	E_a, kJ·mol^{-1}	lnC_E
GaAs	6 H_2O_2 + 94 HBr	14.9 ± 0.2	2.0 ± 0.8
InAs(111)A		12.8 ± 0.1	1.9 ± 0.2
InAs(111)B		2.03 ± 0.3	0.8 ± 0.5
InAs(Sn)		10.5 ± 0.2	1.8 ± 0.5
GaSb		9.6 ± 0.1	1.8 ± 0.4
InSb		51.2 ± 0.3	3.1 ± 0.9
GaAs	10 H_2O_2 + 75 HBr + 15 EГ	15.3 ± 0.1	2.0 ± 0.5
InAs(100)		8.6 ± 0.1	1.6 ± 0.3
InAs(Sn)		14.1 ± 1.0	1.9 ± 0.2
GaSb		6.6 ± 0.1	1.5 ± 0.3
InSb		14.9 ± 0.1	2.1 ± 0.1
GaAs	4 H_2O_2 + 81 HBr + 15 $C_6H_8O_7$	12.8 ± 0.1	1.9 ± 0.1
InAs(100)		16.6 ± 0.1	2.1 ± 0.4
InAs(Sn)		18.1 ± 0.2	2.2 ± 0.7
GaSb		3.8 ± 0.1	1.9 ± 0.3
InSb		18.5 ± 0.1	2.2 ± 0.3
GaAs	10 H_2O_2 + 75 HBr + 15 $C_4H_6O_6$	7.2 ± 0.1	1.6 ± 0.1
InAs(100)		15.9 ± 0.2	2.2 ± 0.6
InAs(Sn)		7.9 ± 0.6	1.7 ± 0.1
GaSb		23.7 ± 0.1	2.5 ± 0.3
InSb		18.1 ± 0.2	2.3 ± 0.6
GaAs	10 H_2O_2 + 75 HBr + 15 $C_3H_6O_3$	6.5 ± 0.1	1.4 ± 0.1
InAs(111)A		23.2 ± 1.0	2.4 ± 0.1
InAs(111)B		25.1 ± 1.6	2.5± 1.4
InAs(Sn)		16.9 ± 0.9	2.3 ± 2.1
GaSb		20.1 ± 0.3	2.4 ± 0.9
InSb		25.8 ± 1.6	2.6 ± 1.3

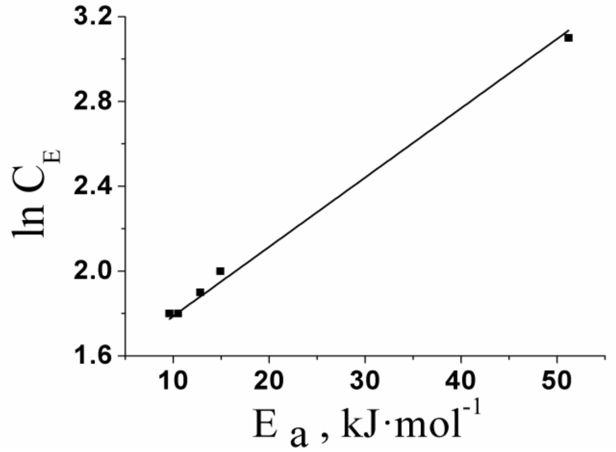

Fig. 1. Dependence of the logarithm of the pre-exponential factor (*ln* C_E) on the apparent activation energy (E_a) for the interaction process of GaAs, InAs, InAs(Sn), GaSb, and InSb with the polishing bromine-releasing solution (in vol.%) 6 H_2O_2 + 94 HBr.

When using bromine-releasing solutions containing an organic component, the dependence of the pre-exponential factor on the calculated apparent activation energy is described by a linear equation:

$$ln\, C_E = (0{,}055 \pm 0{,}002) \cdot E_a + (1{,}196 \pm 0{,}038) \qquad (7)$$

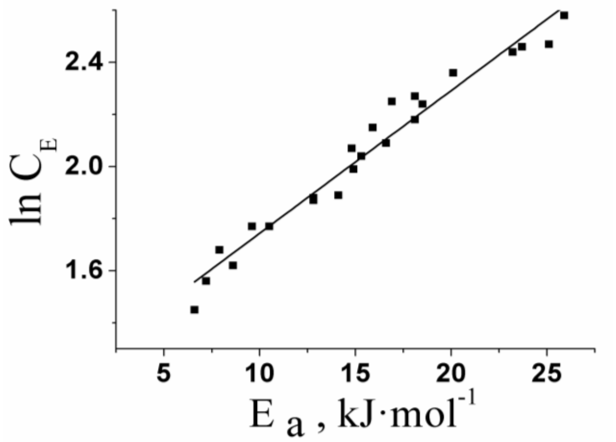

Fig. 2. Dependence of the logarithm of the pre-exponential factor (*ln* C_E) on the apparent activation energy (E_a) for the interaction process of GaAs, InAs, InAs(Sn), GaSb, and InSb with bromine-releasing etchants based on H_2O_2–HBr–solvent solutions.

It can be seen that for the binary bromine-releasing system H_2O_2–HBr, the kinetic compensation effect is less pronounced (indicated by a smaller slope of the line) due to the presence of a relatively small amount of active components in the polishing etching mixture, such as free bromine and bromide ions, which reduces the entropic factor of the chemical interaction process. The existence of the compensation effect also leads to a reduction in the activation energy of the polishing process with the bromine-releasing solution, with a maximum E_a of 15 kJ·mol^{-1} (except for InSb) compared to bromine-releasing etching mixtures containing an organic solvent, where the maximum E_a reaches 25.8 kJ·mol^{-1}. The increase in activation energy is attributed to additional activation processes occurring in the solution related to the presence of the organic component, which can partially interact with bromine, form complexes with the reaction products, and adsorb onto the surface of III-V semiconductor materials [17].

No influence of the chemical composition of the III-V semiconductor materials on the linear equation describing the compensation dependence was observed, likely due to the similar crystal structures and the significant impact of the entropic factor during the transition of semiconductor atoms from the solid to the liquid phase at the chemical etching, which is limited by diffusion stages of the process. Additionally, no influence of the crystallographic orientation of the semiconductor material on the compensation effect was detected.

Conclusions

The presence of the compensation effect enables the purposeful development of technological regimes for surface formation of GaAs, InAs, GaSb, and InSb semiconductor compounds with tailored composition as well as desired physicochemical and geometric parameters. Identification of the heterogeneous processes occurring at the solid–liquid interface provides a basis for designing etching compositions based on the H_2O_2–HBr system with improved performance characteristics. The introduction of an organic solvent into the H_2O_2–HBr etching mixture facilitates the transition of the InSb dissolution process from the kinetic to the diffusion-controlled regime.

The presence of compensation dependence also provides a thermodynamic justification for effective chemical treatment methods of semiconductor materials, offering a theoretical foundation and practical means for regulating the chemical dissolution process by optimizing temperature conditions, incorporating additional components into the etching composition, and controlling other parameters of the etching process.

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Компенсаційний ефект в кінетиці хімічної обробки GaAs, InAs, GaSb та InSb бромвиділяючими травильними композиціями на основі розчинів H_2O_2–HBr з метою вибору ефективних методик їх хімічного полірування

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В роботі досліджено кінетичні залежності хіміко-динамічного травлення напівпровідникових сполук GaAs, InAs, GaSb та InSb в поліруючих травильних сумішах H_2O_2–HBr та H_2O_2–HBr–органічний розчинник. Використовуючи температурні залежності кінетики полірування досліджуваних напівпровідників в розчинах на основі системи H_2O_2–HBr розраховано уявні енергії активації хімічного розчинення поверхні кристалів та значення передекспоненційного множника в рівнянні Арреніуса. Встановлено існування кінетичного компенсаційного ефекту між енергією активації та значенням передекспоненційного множника в гетерогенному процесі полірування поверхні GaAs, InAs, GaSb та InSb. Показано, що на компенсаційну залежність не впливає органічний розчинник в складі бромвиділяючої травильної композиції, а також хімічна природа напівпровідникового матеріалу типу $A^{III}B^{V}$.

Ключові слова: рівняння Арреніуса, уявна енергія активації, кінетичний компенсаційний ефект.