

EVAPORATION THERMODYNAMICS AND SUBLIMATION OF ALUMINUM TELLURIDE

Burabaeva N.M.*, Volodin V.N., Nitsenko A.V., Tuleutai F. Kh.

"Institute of Metallurgy and Ore Beneficiation" JSC, Satbayev University, Almaty, Kazakhstan

* Corresponding author email: Nuri_eng@mail.ru

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ABSTRACT

The analysis of the researches completed by now showed the lack of information concerning the definition of quantity of the vapor pressure values over the molten and crystalline aluminum chalcogenide. In the work presented here, the saturated vapor pressure over liquid and crystalline aluminum sesquioxide was determined for the first time by the boiling point method (isothermal version). The compound was synthesized from elements, with a purity of 99.99 wt. %, identified by X-ray phase analysis as a monophase Al_2Te_3 was used as a research object. Certain vapor pressure of liquid Al_2Te_3 corresponds to the dependence,

$\ln p_{Al_2Te_3} [Pa] = 18,828 - 11865 \cdot T^{-1}$ vapor pressure over crystalline telluride is

$\ln p_{Al_2Te_3} [Pa] = 19,869 - 13077 \cdot T^{-1}$

Based on the values of saturated vapor, the temperature dependence of the Gibbs free energy of evaporation and sublimation was determined, by differentiating which concerning temperature, the entropies of the condensed phase - vapor transformation were calculated, and then the enthalpy. Thermodynamic functions were as follows: entropy of evaporation of the liquid phase - 60.71 ± 4.08 J/(mol K), enthalpy - 98.65 ± 6.64 kJ/mol; entropy of sublimation of the crystalline phase - 69.37 ± 4.67 J/(mol K), enthalpy - 108.73 ± 7.31 kJ/mol. The low value of entropy of the aluminum telluride transfer to the vapor phase indicates the presence of associates in the vapor and of the congruent character of evaporation and sublimation of Al_2Te_3 indirectly. Defined as the difference between the sublimation enthalpies and evaporation, the enthalpy of aluminum telluride melting was 10.08 ± 0.68 kJ/mol, the entropy calculated similarly was 8.66 ± 0.58 J/(mol K). The data obtained coincide with the thermodynamic values found by other authors by calorimetric methods.

Keywords: aluminum telluride, vapor pressure, melting, evaporation, sublimation

Burabaeva Nurila Muratovna

Information about authors: Candidate of Technical Sciences, Senior researcher, JSC "Institute of Metallurgy and Ore Beneficiation", Almaty, Kazakhstan. ORCID ID: 0000-0003-2183-2239, Email: Nuri_eng@mail.ru, Almaty, Kazakhstan.

Volodin Valerij Nikolaevich

Doctor of technical sciences, professor, doctor of physical and mathematical sciences, professor, Chief scientific employee JSC "Institute of Metallurgy and Ore Beneficiation", Almaty, Kazakhstan. ORCID ID: 0000-0001-9543-5944, Email: volodinv_n@mail.ru

Nitsenko Alina Vladimirovna

Candidate of Technical Sciences, Head of of the laboratory of vacuum processes JSC "Institute of Metallurgy and Ore Beneficiation", Almaty, Kazakhstan. ORCID ID: 0000-0001-6753-0936. Email: nitc@inbox.ru

Tuleutai Farkhat Khanafiyaully

Master of Technical Sciences, Engineer, JSC "Institute of Metallurgy and Ore Beneficiation", Almaty, Kazakhstan, Email: farkhat_kaldybek@mail.ru.

Introduction

A retrospect of seventy years showed a limited number of researches devoted to the physicochemical study of aluminum chalcogenides, compared with similar compounds of other metals [[1],[2],[3],[4],[5],[6],[7]], and the most of these few studies are associated with the aluminum-tellurium system.

Under the equilibrium state diagram [8], there is one compound, aluminum sesquioxide selenide

(Al_2Te_3) in the tin-selenium system in the condensed phase, which melts congruently at temperatures of 895 °C.

The authors of [9] found the heat of formation of aluminum sesquichalcogenides, for a compound with tellurium - 326.3 ± 21 kJ/mol by direct determination of the heat of interaction of metal with chalcogenes in the Berthelot-Roth microbomb.

A close value of the enthalpy of formation of aluminum telluride (-318.8 ± 4 kJ/mol) based on calorimetric measurements was obtained in [10].

As a result of the approximate calculation [11], the heat of gaseous Al_2Te_3 formation was 364 kJ/mol.

Mass spectrometric studies [12] of the composition of the vapor phase over aluminum telluride found the presence of AlTe^+ , Al_2Te^+ , Al_2Te_2^+ , and AlTe_2^+ ions with a low relative intensity and found the enthalpies and entropies of formation of compounds of the indicated composition at a temperature of 1,292K (1,019 °C). The dissociation energy of AlS was determined to be 359.8 ± 12.6 kJ/mol.

A group of authors first determined the enthalpies of formation and melting of alloys with a concentration of up to 70 at. % Those, where the congruent character of melting of Al_2Te_3 was established, and then, using differential thermal analysis [14], the presence of a stratification region of liquid solutions was found.

Mass spectrometric studies and the values of the formation enthalpies obtained in the same study confirmed the presence of two liquid phases.

When studying the pressure and composition of vapor by mass spectroscopic, static, and torsion-effusion methods [15] in the temperature range 538-760 K, it was found that the only component of the gas phase over crystalline Al_2Te_3 is the Te_2 dimer, and the temperature dependence of the dissociation pressure (\bar{p}_{Te_2}) turned out to be equal

$$\lg \bar{p}_{\text{Te}_2} [\text{kPa}] = (5,079 \pm 0,297) - (8270 \pm 152) \cdot T^{-1},$$

where T is the temperature, K.

Partial and integral thermodynamic functions of metals were determined in [16] paper, using the method of measuring the electromotive forces of concentration chains at 700 - 820 K (427 – 547 °C) in the range of alloy compositions of the aluminum-tellurium system up to 20 atom percentte.

When studying phase equilibria and intermediate phases in the Al-Te system, the authors of [17] paper found the presence of a superstructure in the α - Al_2Te_3 compound. The [18] paper gives excess thermodynamic functions in the concentration range 0-60 atom percent tellurium for 1190 K (917 °C), indicating very low values of its thermodynamic activity.

The study [19] is devoted to thermodynamic modeling and optimization of the Al-Te system based on and using previously performed experiments, including [[13], [14]].

No later publications concerning the study of the thermodynamics of the aluminum-tellurium system have been found. The analysis of the research results

outlined above shows a clear lack of Information about the aluminum-tellurium system in respect to the technologies of tellurium purification by physical and physicochemical methods. There is no information regarding the thermodynamics of evaporation and sublimation of aluminum sesquitelluride Al_2Te_3 .

The purpose of this study was to determine the thermodynamic functions of the sublimation of crystalline telluride, its evaporation from the liquid phase, and the melting of the compound.

Research object and methodology

The object of research was aluminum telluride synthesized by fusing the amounts of tellurium (99.99 wt.%) and aluminum (99.99 wt.%) corresponding to the composition of the compound in sealed quartz ampoules, from which air was previously evacuated. Heating was performed at a rate of 100 °C per hour to a temperature of 1000 °C, the melt was kept at this temperature for 12 hours, followed by quenching in water. The alloy was identified by X-ray phase analysis as a monophasic Al_2Te_3 .

When considering the Al-Te system, the congruent nature of melting and the existence of aluminum telluride in solution was noted [13], a very low value of Te activity in the liquid phase at concentrations of 0-60 atom percent, that, apparently, refers to the element obtained as a result of the dissociation of Al_2Te_3 , as well as its low value of the dissociation pressure at a melting temperature of 1168 K (895 °C) - $9.97 \cdot 10^{-3}$ kPa (we calculated from the data [15]).

This fact, as well as the significant value of the free energy of aluminum telluride formation, gives grounds to assume the congruent character of the compound evaporation. thermodynamic characteristics were calculated based on the values of the saturated vapor of Al_2Te_3 .

To determine the vapor pressure values, the boiling point method (isothermal version) was chosen; the method was based on a sharp increase in the evaporation rate when the external pressure and saturated vapor pressure of the investigated component are equal with a decrease in pressure above the melt, which was described in detail by the authors earlier [[20], [21], [22], [23], [24], [25]].

The advantage of the method is that there is no need to establish the molecular composition of the vapor, which introduces significant errors in calculations when determining the saturated vapor

Table -1 – Experimentally determined and calculated values of the saturated vapor of aluminum telluride

Temperature, K	Experiment steam pressure, kPa.	Calculated steam pressure, kPa	Relative error, %	Temperature, K	Experiment steam pressure, kPa.	Calculated steam pressure, kPa	Relative error, % (Δ),
1,473	46.40	47.72	-2.77	1,123	3.73	3.73	± 0.0
	49.06		+2.81		3.47		-6.97
	47.72		± 0.0		4.00		+7.24
1,373	27.52	26.54	+3.69	1,033	1.33	1.35	-1.48
	26.73		+0.72		1.27		-5.93
	25.43		-4.18		1.47		+8.89
1,173	5.73	6.08	-5.76	973	0.67	0.62	8.06
	6.11		-0.49		0.67		+8.06
	6.42		+5.59		0.53		-14.52
							$ \Delta _{\text{average}}=5.13$

pressure by other methods. due to the high aggressiveness of tellurium and telluride concerning metals, quartz was used as the material of the device and the container for the test charge and suspension device material in its high-temperature part. The pressure was measured with a vacuum gauge in mm Hg with subsequent transfer to Pa.

Experimental results and their discussion

The experimental conditions and the values of the vapor pressure of aluminum telluride determined in this case are given in the table 1.

The total measurement error is defined as the sum of the errors of independent measurements: temperature – 1 %, weighing –0.1 %, pressure – 0.5 %, approximation of experimental data –5.13 %, equal to 6.73 %.

The experimentally determined vapor pressure of liquid Al_2Te_3 corresponds to the dependence:

$$\ln p_{Al_2Te_3} [Pa] = 18,828 - 11865 \cdot T^{-1}$$

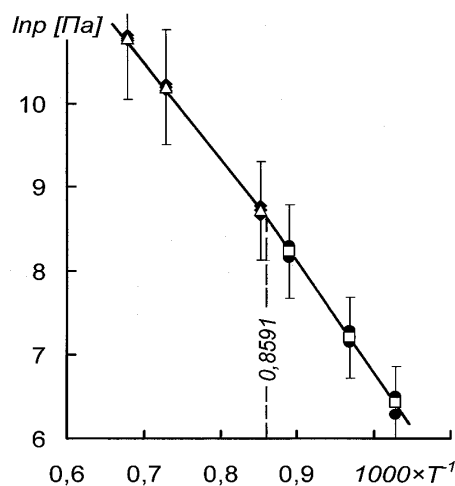
over crystalline telluride –

$$\ln p_{Al_2Te_3} [Pa] = 19,869 - 13077 \cdot T^{-1}$$

The results of the values of the saturated vapor pressure over liquid and crystalline aluminum sesquitelluride in the coordinates $\ln p - T^{-1}$ (figure 1) graphically, by the intersection of linear dependences ($1000 \cdot T^{-1} = 0.8591$) determine the melting point equal to 1,164 K (891 °C) that only differs by 4°C from the one shown on the equilibrium phase (895 °C) diagram [8] and indicates the correctness of the experimental data.

Based on the values of the saturated vapor pressure over aluminum telluride (Al_2Te_3), the thermodynamic functions of evaporation were calculated, which amounted to entropy of the liquid phase – 60.71 ± 4.08 J/(mol K), enthalpy – $98.65 \pm$

6.64 kJ/mol; entropy of sublimation of the crystalline phase - 69.37 ± 4.67 J/(mol K), enthalpy – 108.73 ± 7.31 kJ/mol. The low enthalpy value of the aluminum telluride transfer to the vapor phase indicates the presence of associates in the vapor and, indirectly, the congruent character of Al_2Te_3 evaporation under Truton's rule.



Dark marks are experimental data and light marks are calculated data

Figure 1 - Dependence of the vapor pressure of aluminum telluride on temperature

Based on the simplest relationship: $\Delta H_{mel} = \Delta H_{subl} - \Delta H_{evap}$, where ΔH_{mel} , ΔH_{subl} and ΔH_{evap} are enthalpies of melting, sublimation and evaporation, respectively, the thermodynamic functions of melting of aluminum telluride were determined: enthalpy is 10.08 ± 0.68 kJ/mol, entropy is 8.66 ± 0.58 J/(mol K).

The latter coincides with the data of the study [14], where, using calorimetry, the melting entropy

was determined equal to 8.6 ± 0.9 J/(mol K), and the enthalpy is 10 ± 1 kJ/mol.

Conclusions

As a result of this research, the pressure of saturated vapor over liquid and crystalline aluminum sesquioxide using the boiling point method was determined for the first time and on this grounds, the thermodynamic functions of evaporation and sublimation of Al_2Te_3 were calculated.

The enthalpy of liquid compound vaporization was determined as 98.65 ± 6.64 kJ/mol, of crystalline sublimation was 108.73 ± 7.31 kJ/mol; the evaporation entropy of telluride was found to be equal to 60.71 ± 4.08 J/(mol K), the sublimation entropy was 69.37 ± 4.67 J/(mol K).

The low value of entropy indicates the presence of associates in the vapor and the congruent character of evaporation and sublimation. The melting functions determined in this study: entropy – 8.6 ± 0.9 J/(mol K), enthalpy – 10 ± 1 kJ/mol, coincide with the results of determinations performed with the calorimetric method.

The obtained values of the functions will supplement the currently missing information in the thermodynamic database.

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Алюминий теллуридінің булану және сублимациясының термодинамикасы

Бурабаева Н.М., Володин В.Н., Ниценко А.В., Тулеутай Ф.Х.

«Металлургия және кен байыту институты» АҚ, Сәтбаев университеті, Алматы, Қазақстан

ТҮЙІНДЕМЕ

Осы уақытқа дейін жүргізілген зерттеулерді талдау нәтижесінде балқытылған және кристалды алюминий халкогенидінің бу қысымының мәндерін анықтауға қатысты ақпарат жоқ екені анықталды. Ұсынылған жұмыста қайнау нүктелері әдісімен (изотермиялық нұсқа) алғаш рет қаныққан будың сұйық және кристалды алюминий сесквителлуридіне қысымы анықталды. Зерттеу нысаны ретінде элементтерден синтезделген тазалығы 99,99 мас. %, Al_2Te_3 монофаза ретінде рентген-фазалық талдау арқылы анықталған қосылыс қолданылды. Сұйық Al_2Te_3 белгілі бу қысымы:

$\ln p_{Al_2Te_3} [Па] = 18,828 - 11865 \cdot T^{-1}$ - тәуелділікке сәйкес келеді, кристалды теллурид

үстіндегі бу қысымы: $\ln p_{Al_2Te_3} [Па] = 19,869 - 13077 \cdot T^{-1}$ тең. Қаныққан будың мәндеріне сүйене

отырып, Гиббстің булану мен сублимацияның бос энергиясының температураға тәуелділігі, оны температураға қатысты дифференциалдау арқылы конденсацияланған фазаның энтропиялары – будың түрленуі алдымен есептеледі, содан кейін энтальпия. Термодинамикалық функциялар келесі мәндерді құрады: сұйық фазаның булану энтропиясы - $60,71 \pm 4,08$ Дж/(моль·К), энтальпия – $98,65 \pm 6,64$ кДж/моль; кристалды фазаның сублимация энтропиясы – $69,37 \pm 4,67$ Дж/(моль·К), энтальпиясы – $108,73 \pm 7,31$ кДж/моль. Алюминий теллуридінің бу фазасына өту энтропиясының төмен мәні буда ассоциаттардың болуын және жанама түрде Al_2Te_3 булануы мен сублимациясының конгруэнтті сипатын көрсетеді. Сублимация және булану энтальпияларының айырмашылығы ретінде анықталған алюминий теллуридінің балқу энтальпиясы $10,08 \pm 0,68$ кДж/моль, осыған ұқсас әдіспен есептелген энтропия $8,66 \pm 0,58$ Дж/(моль·К) болды. Алынған деректер калометриялық әдістермен басқа авторлар тапқан термодинамикалық мәндермен сәйкес келеді.

Түйін сөздер: алюминий теллуриді, бу қысымы, балқу, булану, сублимация, энтропия, энтальпия.

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Бурабаева Нурила Муратовна	Авторлар туралы ақпарат: Техника ғылым кандидаты, «Металлургия және байыту институты» АҚ, Алматы, Қазақстан. ORCID идентификаторы: 0000-0003-2183-2239 Электрондық поштасы: Nuri_kaz@mail.ru
Володин Валерий Николаевич	Техника ғылымдарының докторы, профессор, физика-математика ғылымдарының докторы, профессор, «Металлургия және байыту институты» АҚ бас ғылыми қызметкері, Алматы, Қазақстан. ORCID идентификаторы: 0000-0001-9543-5944 Электрондық поштасы: volodinv_n@mail.ru
Ниценко Алина Владимировна	Техника ғылымдарының кандидаты, «Металлургия және байыту институты» АҚ вакуумдық процестер зертханасының меңгерушісі, Алматы қ., Қазақстан. ORCID идентификаторы: 0000-0001-6753-0936. Электрондық поштасы: nitc@inbox.ru
Тулеутай Фархад Ханафияұлы	Магистр, инженер, «Металлургия және байыту институты» АҚ, Алматы, Қазақстан, Электрондық поштасы: farkhat_kaldybek@mail.ru.

Термодинамика испарения и сублимации теллурида алюминия

Бурабаева Н.М., Володин В.Н., Ниценко А.В., Тулеутай Ф.Х.

АО «Институт металлургии и обогащения», Satbayev University, Алматы, Казахстан

<p>Поступила: 15 июня 2021 Рецензирование: 07 ноября 2021 Принята в печать: 16 февраля 2022</p>	<p>АННОТАЦИЯ</p> <p>В результате анализа выполненных к настоящему времени исследований установлено отсутствие сведений, касающихся определений величин давления пара над расплавленным и кристаллическим халькогенидом алюминия. В представленной работе впервые методом точек кипения (изотермический вариант) определено давление насыщенного пара над жидким и кристаллическим сесквителлуридом алюминия. В качестве объекта исследования использовано синтезированное из элементов, чистотой 99,99 мас. %, соединение, идентифицированное рентгенофазовым анализом как монофаза Al_2Te_3. Определенное давление пара жидкого Al_2Te_3 соответствует зависимости: $\ln p_{Al_2Te_3}[Па] = 18,828 - 11865 \cdot T^{-1}$, давление пара над кристаллическим теллуридом - $\ln p_{Al_2Te_3}[Па] = 19,869 - 13077 \cdot T^{-1}$. На основании величин насыщенного пара определена температурная зависимость свободной энергии Гиббса испарения и сублимации, дифференцированием которой по температуре рассчитаны вначале энтропии превращения конденсированная фаза – пар, а затем энтальпия. Термодинамические функции составили величину: энтропия испарения жидкой фазы – $60,71 \pm 4,08$ Дж/(моль·К), энтальпия – $98,65 \pm 6,64$ кДж/моль; энтропия сублимации кристаллической фазы – $69,37 \pm 4,67$ Дж/(моль·К), энтальпия – $108,73 \pm 7,31$ кДж/моль. Малая величина энтропии перевода теллурида алюминия в паровую фазу свидетельствует о наличии ассоциатов в паре и косвенно – о конгруэнтном характере испарения и сублимации Al_2Te_3. Определенная как разница энтальпий сублимации и испарения энтальпия плавления теллурида алюминия составила величину $10,08 \pm 0,68$ кДж/моль, энтропия, рассчитанная аналогично, – $8,66 \pm 0,58$ Дж/(моль·К). Полученные данные совпадают с термодинамическими величинами, найденными другими авторами калориметрическими методами.</p> <p>Ключевые слова: теллурид алюминия, давление пара, плавление, испарение, сублимация</p>
Бурабаева Нурила Муратовна	Информация об авторах: Кандидат технических наук, АО "Институт металлургии и обогащения", Алматы, Казахстана. ORCID ID: 0000-0003-2183-2239, Email: Nuri_eng@mail.ru
Володин Валерий Николаевич	Доктор технических наук, профессор, доктор физико – математических наук, профессор, главный научный сотрудник АО "Институт металлургии и обогащения", Алматы, Казахстан. ORCID ID: 0000-0001-9543-5944, Email: volodinv_n@mail.ru
Ниценко Алина Владимировна	Кандидат технических наук, заведующая лабораторией вакуумных процессов АО "Институт металлургии и обогащения", Алматы, Казахстан. ORCID ID: 0000-0001-6753-0936. Email: nitc@inbox.ru
Тулеутай Фархад Ханафияұлы	Магистр, инженер, АО "Институт металлургии и обогащения", Алматы, Казахстан. Email: farkhat_kaldybek@mail.ru.

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