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Silicon Refining by Growing Crystallites in a Hypereutectic Melt of Aluminum with Silicon

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ABSTRACT

Silicon is an essential chemical element that plays a very important role in life support on our planet. There is no single area of life on Earth where this most common chemical element is not present; its content is 27-30% of the mass of the Earth's crust. The most common form of its presence in the earth's crust is silicon dioxide, that is, silica. Silica is the main raw material source for semiconductor silicon for modern electrical engineering (production of diodes, transistors, photocells, and integrated circuits). Silicon is widely used in special materials science (alloying special steels, refining melts, and producing aluminum-silicon alloys (silumins). Reducing the cost of high-purity silicon can be achieved by reducing the temperature of the refining process of technical silicon. This scientific work examines the implementation of refining silicon in eutectic silicon melts with aluminum. Crystallization of silicon in hypereutectic melts below the liquidus temperature of the Si-Al phase equilibrium system occurs through the formation of silicon crystallization nuclei from clusters on the surface of the crystallizer - electrodes (rods) made of silicon with a temperature, which is below the temperature of the hypereutectic melt liquidus. The article describes methods for obtaining high-purity silicon and provides scientific justification for the implementation of such a task. The proposed method for producing high-purity silicon has so far been little studied and is of scientific and economic interest, since it can be carried out at relatively low temperatures, for example below 900°C. An important feature of eutectic melts is the stoichiometric content of components, which was the reason for the assumption of the molecular structure of eutectic melts. Eutectic molecules are, in our opinion, compounds of silicon and aluminum clusters.

Keywords: silicon, aluminum, cluster, crystallite, liquation, crystallization.

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Introduction

Silicon is the most important chemical element on our planet. There is not a single area of life on the Earth where this most common component is present, its content amounts to 27-30% of the mass of the Earth's crust [[1], [2], [3], [4]]. The most common form is silicon dioxide–silica. Silica is the main raw material source for semiconductor silicon for modern electrical engineering (production of diodes, transistors, photocells, and integrated circuits). Silicon is widely used in special materials science (alloying special steels, refining melts, and producing aluminum-silicon alloys (silumins). The growing need for high-purity silicon for photovoltaic energy converters, caused by the development of "Green Energy", served as the basis for the search and the development of effective technologies for refining metallurgical silicon to the level of "Solar" quality silicon, that is, with a Silicon content of 99.9999%. Without such a degree of purification of silicon from impurities, it is impossible to obtain photovoltaic energy converters with a high efficiency for efficient solar batteries.

Silicon refining using a hypereutectic melt of aluminum and silicon is a process that is used to remove impurities and improve the quality of silicon, especially in the production of silicon single crystals for the semiconductor industry. Here's how it works:

1. Preparation of raw materials: First it is necessary to prepare the raw materials. This may be metal silicon, which contains impurities such as aluminum, iron, and other metals. A mixture of aluminum and silicon is also suitable.

2. Melting: A mixture of silicon and aluminum is heated to a temperature beyond the melting point of silicon. This ensures the formation of a hypereutectic liquid, which can interact with silicon, but not aluminum.

3. Separation: In a hypereutectic liquid, silicon dissolves while aluminum remains unchanged. This allows aluminum and other impurities to be removed from the silicon. The separation occurs due to differences in the solubility of aluminum and silicon in a given liquid.

4. Cooling and crystallization: After separation, the silicon begins to crystallize within the liquid. This can happen by slowly cooling the mixture. Silicon crystals can be produced as single crystals or polycrystals, depending on the process.

5. Silicon extraction and processing: The resulting silicon crystals are then removed from the melt and further processed and purified to improve their quality.

This process produces purer silicon suitable for semiconductors and other high-tech industries. It is important to note that it can be resource and energy-intensive, and its effectiveness depends on the exact conditions and process parameters.

Existing technologies and methods for purifying silicon from impurities, such as refining by blowing the silicon melt with oxygen, zone melting, electrolysis in molten salts, sublimation of silicon monoxide and its reduction with magnesium and others do not provide the required degree of purification from impurities, are characterized as environmentally unsafe and energy-intensive. The Czochralski Method has proven itself to be quite effective [[2], [3], [4], [5], [6], [7], [8], [9]]. This refining method, although very expensive in terms of production costs, is still considered the most common in the production of semiconductor silicon [[10], [11], [12], [13], [14], [15]].

Methods

The experiments were carried out according to the following technological scheme:

1. Preparation of a hypereutectic alloy of silicon with aluminum, implemented in the following sequence:

1.1 melting silicon Kr1 in an induction furnace with a protective atmosphere (Nitrogen or carbon dioxide).

1.2 aluminum of Al1 grade (electrical) was loaded into a silicon melt at a temperature of 1415-1450°C, its concentration was adjusted to 20% (mass). A melt was obtained in which, upon cooling, crystallization began with the formation of silicon clusters.

1.3 when immersing a rotating rod made of pure silicon heated to a melt temperature of 660-690°C into the melt, the rotation speed was selected experimentally and amounted to 5-10 min^{-1}.

The process of silicon crystallization on the crystallizers was monitored visually; the duration of the crystallizer's stay in the melt did not exceed 30 minutes.

2. Analysis of the elemental composition of the resulting refined silicon was carried out using the X-ray fluorescence method on a scanning electron microscope with an analyzer at the Research Regional Engineering Laboratory "Structural and Biochemical Materials" at M. Auezov South Kazakhstan University, equipment used: Spekord V-80 and Superprobe 733-SCX.

Research Results and Discussion

This work studied the possibility of refining silicon in eutectic melts using the features of liquation processes and the limited solubility of melt components in the crystallizing phases of the eutectic, under the conditions of the beginning of silicon crystallization in the eutectic melt. The Silicon–Aluminum system with eutectic (Al 88% + Si 12%) melting at 577°C was chosen as the base material, Fig.1, [9].

This process of obtaining high-purity silicon has not yet been well studied and is of scientific and economic interest, since it can be carried out at relatively low temperatures, for example below 900°C. An important feature of eutectic melts is the stoichiometric content of components, which was the reason for the assumption of the molecular structure of eutectic melts [16].

Eutectic molecules are, in our opinion, compounds of silicon and aluminum clusters. Clusters of these components can be imagined if consider the crystal structure of aluminum and silicon (Fig.2).

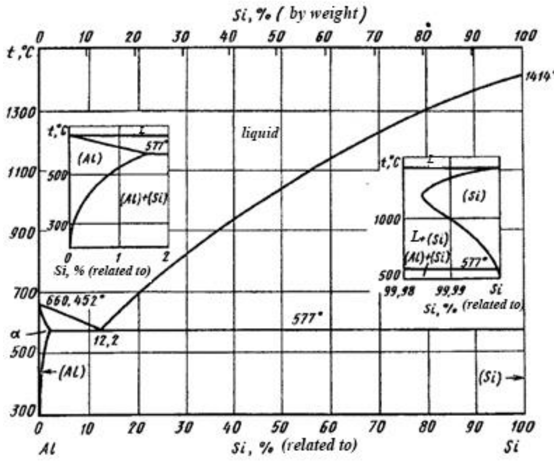
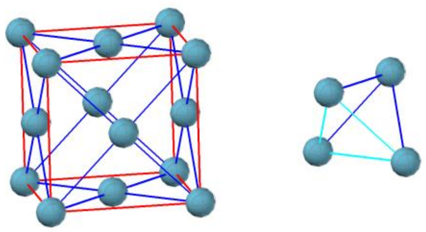
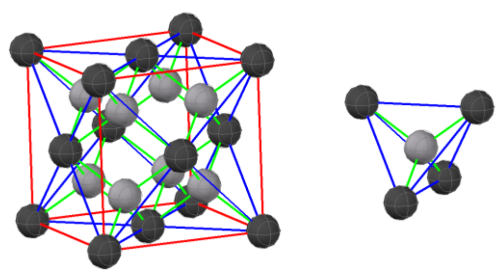


Figure 1 - Aluminum-silicon phase equilibrium diagram [9]



Crystal structure of aluminum
Atomic structure of cluster
a)



Crystal structure of silicon
Atomic structure of cluster
b)

Figure 2 - Crystal structure of aluminum and silicon atoms and atomic structure of clusters

Clusters in the melt are grouped into complex molecules according to the chemical composition of the eutectic 12.2% Si and 87.8% Al (mass per cent) [[16], [17], [18], [19]] (Fig.3).

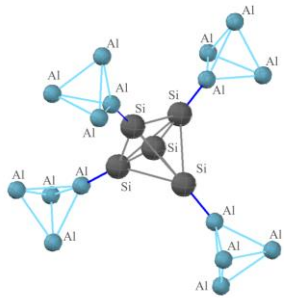


Figure 3 - Structure of a complex molecule of eutectic aluminum-silicon melt

This arrangement of the atomic eutectic melt structure ensures the density (specific gravity) of the eutectic melt close to the density of the crystallized eutectic alloy [[20], [21], [21], [22], [23], [24], [25]]. Figure 3 offers the reader the structure of the molecule of a eutectic aluminum-silicon melt. The main feature of the structure is that it consists of complex molecules of clusters of silicon atoms and clusters of aluminum atoms. The possibility of forming such complex molecules is explained by the geometric similarity of the clusters. The next reason for the formation of complexes is the almost identical density of the substance in the liquid and solid-crystalline states. The distinctive structure of the silicon cluster lies in the size of interatomic distances and the presence of the silicon atom with electronic bonds involved inside the cluster. This complicates the formation of crystallization nuclei and increases the stability of the liquid eutectic mixture of complex molecules and individual clusters of silicon and aluminum. Therefore, in eutectic melts of silicon and aluminum at temperatures below the liquidus temperature, clusters of silicon atoms are formed without admixtures of other atoms.

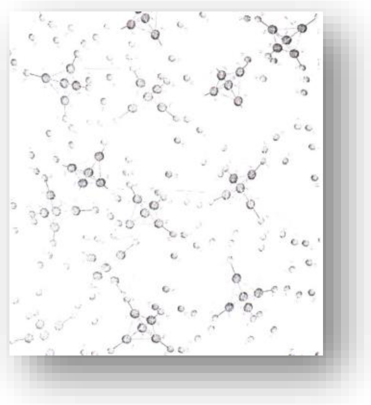


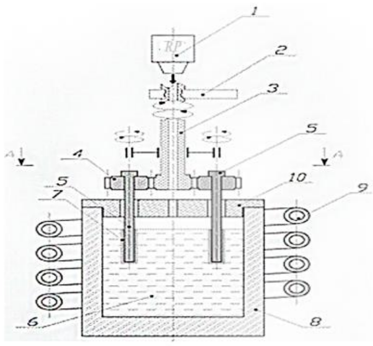
Figure 4 - Scheme of the atomic structure of a eutectic melt of silicon with aluminum (complex eutectic molecules and excess silicon clusters)

This is confirmed by the results of the scientific work of the authors, for example, at a temperature close to 900°C, crystallizing silicon has a purity of 99.99% (see an inset in the diagram in Fig. 1) [[16], [17], [20]].

By creating conditions for silicon crystallization, immersing crystallizer rods in the melt at a temperature 5-10°C below the liquidus temperature, we will ensure the deposition of silicon clusters on the surface of the silicon crystallizer. Silicon crystallization nuclei – silicon clusters in the eutectic melt provide a high rate of crystallization of high-purity polycrystalline silicon. If a crystallizer is used from a single crystal, it is possible to obtain monocrystalline silicon, but this is not a technically simple task and requires additional research. To regulate the rate of silicon crystallization, the surface temperature of the crystallizer is maintained within a specified range. Figure 5 shows a diagram of the refining installation and a photograph of a crystallizer with a crystallized layer of high-purity silicon. Complex molecules of silicon and aluminum clusters provide a high rate of crystallization; excess clusters in the hypereutectic melt crystallize on the surface of the crystallizer, maintaining a high degree of purity from harmful impurities.

For the continuity of the process, the melt is replenished with silicon clusters by dissolving the electrodes of the Aluminum – Silicon alloy and removing melts enriched with impurities from the refining zone. A flow process of hypereutectic melt through the crystallization zone of high-purity silicon is also provided. In this case, the secondary product can be used for casting parts from silumin. The main idea behind the new refining of silicon at temperatures below 900°C and above 5770°C will be widely used and useful for the production of products and alloys from silumin as an additional and efficient production of high-purity silicon.

Two options were tested: 1 – The process in a constantly renewed eutectic melt, that is, to grow high-frequency Silicon clusters on a crystallizer in a flow of always “new” eutectic melt. Growing crystalline silicon from a silumin melt – for a eutectic alloy of silicon with aluminum, ensures the filtration of impurities by the eutectic melt according to a mechanism similar to the process of zone melting [[9], [26]]. Impurities are pushed into the liquid phase, and the surface of the growing crystals on a rotating crystallizer is covered with clusters that form high-purity silicon crystallites. The basis for this process is the limited solubility of aluminum in silicon, which is confirmed by the authors [[16], [17], [20]]. Aluminum clusters act as a sorbent for impurities harmful to silicon. Movement of the surface of the crystallizing rod in the melt (rotation of the crystallizer rod) eliminates the accumulation of impurity atoms in the crystallization zone.



a)

b)

Figure 5 - Diagram of a laboratory refining installation

a) photograph of crystallizers with frozen refined silicon
b) 1 – radiation pyrometer; 2 – clutch with a manual drive lever; 3 – shaft gear with a through hole for transmitting rays of electromagnetic waves of light flux from the mirror of the melt bath to the radiation pyrometer; 4 – satellites (gears) for rotating the crystallizer rods; 5 – crystallizer rods; 6 – eutectic melt; 7 – frozen layer of refined silicon crystals; 8 – graphite crucible; 9 – inductor

The problem of removing accumulating impurities in the eutectic melt from the crystallization zone of refined silicon in crystallizers to ensure the minimum permissible concentration of impurities in the eutectic melt requires additional research and refinement of the technology [[27], [28], [29], [30], [31], [32], [33]].

The research results are shown in Table 1. The results show a significant increase in the degree of purity of the resulting polycrystalline silicon: from the original 97-98% Si to 99.95-99.99% Si. The degree of purity – the content of impurities in the polycrystalline silicon obtained after refining – is significantly influenced by:

- temperature range of the crystallization process on the crystallizer rod (Fig.4),
- rotation speed of the crystallizer rod, which ensures the renewal of the melt at the boundary between the crystallizer and the melt, excluding an increase in the concentration of impurities pushed

aside by growing crystallization centres into the liquid phase – eutectic melt.

Replacing the composition of the crystallizer bath and changing the fuse – the silicon rod coated with frozen high-purity silicon crystals – are very important factors influencing the quality of refining. These issues require careful study and research funds.

Table 1 - Elemental composition of metallurgical silicon and the resulting high-purity silicon by refining by crystallization from the eutectic melt

Sample name	Impurity content, % e				Note
	Ca	**Fe**	**P**	**B**	**Si**
MG-Si	0.016	0.28	0.0021	0.0030	99.69
Refining melt (original)	0.041	0.073	0.0007	0.0017	Melt: Si 20-30%, base: Al
Refining silicon	0.0008	Not detected	0.0003	0.0009	99.998 % Si
Refining melt (spent)	0.54	0.455	0.0615	0.0080	1.578% impurity

Conclusions

Conducted studies have shown the promise of using eutectic melts of silicon with aluminum as efficient in energy saving and relatively low process temperatures – below 900°C.

The possibility of using a similar process for growing silicon single crystals using a method similar to the Czochralski method cannot be ruled out, but this requires extensive research and funds to carry it out.

Research in this work revealed the need to study the refining ability of other eutectics with silicon and to create a technological process for refining silicon to silicon of "solar" quality using the selectivity of melts to absorb harmful impurities in high-purity silicon.

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***CRediT author statement*:** **A. Protopopov:** Conceptualization, Methodology, Software, Data curation, Writing-Original draft preparation. **M. Protopopov:** Visualization, Investigation. **Y. Suleimenov:** Supervision. **R. Altynbekov:** Software, Validation, Writing- Reviewing and Editing.

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Алюминий мен кремнийдің эвтектикадан кейінгі балқымасында кристаллиттерді өсіру арқылы кремнийді тазарту

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Сараптамадан өтті: *18 наурыз 2024*
Қабылданды: *15 шілде 2024*

ТҮЙІНДЕМЕ
Кремний-біздің планетамыздағы тіршілікті қамтамасыз етуде өте маңызды рөл атқаратын маңызды химиялық элемент. Бұл ең көп таралған химиялық элемент жердің барлық аймағында бар, оның мөлшері жер қыртысының массасының 27-30% құрайды. Оның жер қыртысындағы ең көп тараған қосылысы - кремний диоксиді, яғни кремнезем. Кремний диоксиді қазіргі электротехника үшін жартылай өткізгіш кремнийді алудың негізгі шикізат көзі болып табылады (диодтар, транзисторлар, фотоэлементтер, интегралдық схемалар өндірісі). Кремний арнайы материалтану саласында кеңінен қолданылады (арнайы болаттарды легирлеу, балқымаларды тазарту, алюминий-кремний қорытпаларын (силуминдер) алу). Өте таза кремнийдің құнын төмендетуге техникалық кремнийді тазарту

процесінің температурасын төмендету арқылы қол жеткізуге болады. Бұл ғылыми жұмыста кремнийдің алюминиймен эвтектикалық балқымаларында кремнийді тазарту қарастырылады. Si-Al фазалық тепе-теңдік жүйесінің ликвидус температурасынан төмен эвтектикалық балқымалардағы кремнийдің кристалдануы кристаллизатордың бетіндегі кластерлерден кремнийдің кристалдану эмбриондарының - кремнийдің электродтарының (шыбықтарының) түзілуі арқылы жүреді. Мақалада өте таза кремний алу әдістері сипатталған және осындай әдістерді жүзеге асырудың ғылыми негіздемелері келтірілген. Өте таза кремнийді алудың ұсынылған әдісі әлі аз зерттелген және ғылыми және экономикалық қызығушылық тудырады, өйткені оны салыстырмалы түрде төмен температурада, мысалы, 900oC-тан төмен температурада жүзеге асыруға болады. Эвтектикалық балқымалардың маңызды белгісі – олардың молекулалық құрылымын болжауға себеп болған компоненттердің стехиометриялық құрамы. Эвтектикалық молекулалар, біздің ойымызша, кремний мен алюминий кластерлерінің қосылыстары.

Түйін сөздер: кремний, алюминий, кластер, кристаллит, ликвация, кристалдану.

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Рафинирование кремния выращиванием кристаллитов в заэвтектическом расплаве алюминия с кремнием

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АННОТАЦИЯ

Кремний – важнейший химический элемент, играющий очень важную роль для жизнеобеспечения на нашей планете. Нет ни одной области жизни на Земле, где бы ни присутствовал этот, самый распространенный химический элемент, его содержание составляет 27-30% от массы земной коры. Наиболее распространенная форма его присутствия в земной коре – это диоксид кремния, то есть кремнезем. Кремнезем — это основной сырьевой источник получения полупроводникового кремния для современной электротехники (изготовление диодов, транзисторов, фотоэлементов, интегральных схем). Широкое применение кремний получил в специальном материаловедении (легирование специальных сталей, рафинирование расплавов, получение алюминий-кремниевых сплавов (силуминов). Снижение себестоимости высокочистого кремния может быть обеспечено снижением температур процесса рафинирования технического кремния. В данной научной работе рассматривается осуществление рафинирования кремния в эвтектических расплавах кремния с алюминием. Кристаллизация кремния в заэвтектических расплавах ниже температуры ликвидус системы фазового равновесия Si-Al происходит формированием зародышей кристаллизации кремния из кластеров на поверхности кристаллизатора - электродов (стержней) из кремния с температурой ниже температуры ликвидус за эвтектическим расплавом. В статье расписаны спосбы получения кремния высокочистого и приводятся научные обоснования реализации такой задачи. Предлагаемый метод получения высокочистого кремния пока мало изучен и представляет научный и экономический интерес, так как может осуществляться при относительно низких температурах, например ниже 900oC. Важным признаком эвтектических расплавов является стехиометрическое содержание компонентов, что послужило причиной для предположения о молекулярном строении эвтектических расплавов. Молекулы эвтектики представляют собой, по нашему мнению, соединения кластеров кремния и алюминия.

Ключевые слова: кремний, алюминий, кластер, кристаллит, ликвация, кристаллизация.

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