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The method of production of metal silicon solution, the method of production of metal silicon out of the solution and the metal silicon received on the basis of these methods, the method of production of ceramic materials and the ceramic material received on the basis of this method

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A.D. Korchagin
General Director

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(54) THE METHOD OF PRODUCTION OF METAL SILICON SOLUTION, THE METHOD OF PRODUCTION OF METAL SILICON OUT OF THE SOLUTION AND THE METAL SILICON RECEIVED ON THE BASIS OF THESE METHODS, THE METHOD OF PRODUCTION OF CERAMIC MATERIALS AND THE CERAMIC MATERIAL RECEIVED ON THE BASIS OF THIS METHOD
(57) The invention refers to the material production technology, namely to the technology of production of polycrystalline silicon and its chemical compounds – carbide and nitride – out of natural siliceous concentrates. The essence of invention consists in the method of reception of metal silicon solutions, which includes separation of silicon out of silicon-containing compounds mainly out of oxides, its transportation by the binding carrier and dissolution of it in metal melts. The metal silicon thus is received by re-crystallizing raw material in kind of metal silicon solutions under the lowered environmental pressures. The received metal silicon, which is characterized by impurities containing in it, dispersity and specific surface of polycrystalline conglomerates, differs by high purity and low cost price. The ceramic materials are received on the basis of metal silicon by its thermo-chemical interacting with nitrogenous and carbonaceous substances. The received ceramic materials are characterized by their law impurity level, high dispersity and specific surface of their polycrystalline conglomerates, a well as by an optimal for a number of practical cases content of their phase composition. As a whole the offered methods of production of polycrystalline silicon and its compounds – carbide and nitride – out of natural siliceous concentrates allows receiving metal silicon and ceramic material in kind of silicon nitride and/or carbide and providing high economic and ecological efficiency (5 formulas, tab. 1).

The invention refers to the sphere of material production technology, namely to the technology of reception of polycrystalline silicon and its chemical compounds – carbide and nitride – out of natural siliceous concentrates.

There are known the methods of reception of polycrystalline silicon by thermo-chemical reduction of silicon oxides by electrolysis out of melts containing oxidic silicon compounds and salt halogenides (O.V. Pelevin, V.P. Grishyn Production of Semiconductors for Perspective Solar Energy Transformers. - M., ВИНИТИ, The Results of Science and Engineering, series Metallurgy of Non-Ferrous Metals, vol. 19, 1989, pp. 3-48; E.S. Falkevitch, E.S. Pulner, I.F. Chervonnyi and othes The Technology of Semiconductor Silicon. – M., Metallurgy, 1992, p. 408; A.K. Agraval, A.E. Austin //J. Electrochem. Soc., 1980, vol. 127, № 3, p. 117; R.V. Chernov, V.M. Moshenko, N.M. Storchak Electrolyte for Getting Metal Silicon out of Melts. A.C. USSR 460326 declared on 10.06.1973 № 1943090/22-1) with the further synthesis of carbide and nitride out of simple substances (T.Ya. Kosolapova and others The Non-Metallic High-Melting Compounds. – M., Metallurgy, 1986).

There is known the method of silicon separation out of siliceous compounds or metallurgical silicon with transporting it by the binding carrier on the base of chlorine in kind of $SiCl_4$, $SiHCl_3$ with the further hydrogenating and pyrolysis of silane or vapour-phase reduction of $SiCl_4$ and zinc by the reaction (I.V. Grankov Production of Silicon Abroad. /Обозинформ. Issue 1 – M., ЦНИИцветмет, 1983, p. 38):

$$SiCl_4 + 2Zn \rightarrow Si + 2ZnCl_2.$$

There are also known dissolution of metal calcium in the melted zinc at fluorine production by electrolysis of tetrafluoroborate melts containing calcium fluoride (G. Mamontov and T.M. Laher – Production of Fluorine by the electrolysis of Calcium Fluoride – Containing Tetrafluoroborate Melts. //J.

Electrochem. Soc., 1989, vol. 136, № 3, pp. 673-676) and the method of silicon transportation by the binding carrier on the base of chlorine and dissolution of the electrolytically received metal in the melt of fluid zinc cathode (I.V. Grankov Production of Silicon Abroad. /Обозинформ. Issue 1, - M., ЦНИИцветмет, 1983, p. 38; G. Mamontov and T.M. Laher Production of Fluorine by the Electrolysis of Calcium Fluoride – Containing Tetrafluoroborate Melts. //J. Electrochem. Soc. 1989, vol. 136, № 3, pp. 673-676).

There are known the method of metal production by distilling off impurities under the lowered pressures (A.N. Zelikman, G.A. Meerson - M., Metallurgy, 1973, p. 603) and the method of vacuum separation of the reactionary mass at production of metal titanium (K.A. Bolshakov Chemistry and the Technology of Rare and Dissipated Elements. - M., The Higher School, 1976, p. 105).

The metal silicon of solar grade is known (O.V. Pelevin, V.P. Grishyn Production of Semiconductors for the Perspective Solar Energy Transformers. – M., ВИНИТИ, The Results of Science and Engineering, series Metallurgy of Non-Ferrous Metals, vol. 19, 1989, pp. 3-48).

There are known the methods of production of non-metallic high-melting compounds (T.Ya. Kosolapova and others The Non-Metallic High-Melting Compounds. – M., Metallurgy, 1986) and the method of production of ceramic materials on the base of the metal silicon by its thermal interacting with carbonaceous and/or nitrogenous substances (G.G. Gnesin The Non-Oxygen Ceramic Materials. – Kiev, Engineering, 1987).

The ceramic materials are known (O.P. Kulik, S.N. Matsegora, L.M. Averbukh Feed Stock for Production of the Constructional Ceramics – Requirements and Properties //Engineering. Ser. Equipment, materials, processes, 1988, issue 2, p. 85; Patent. 4368180 USA of 11.01.83; J. Soc. Mater. Sci., 1986, vol. 21, № 4, pp. 1448-1456; M.E. Kuimova, A.A. Maksimov Production and Application of New Constructional Ceramic Materials. //New Materials and Technologies. Issue 18, M., ВНТИЦентр, 1986). Their disadvantage is poor reproducibility of the properties because of rather high content of metal impurities, which are the concentrates of voltage in ceramic products.

The common disadvantage of the specified methods and materials is a low quality of the received silicon, its carbide and nitride, and their defecation represents the complex and expensive engineering problem.

The problem solved by the present invention is a production of highly pure powders of the polycrystalline silicon, its carbide and nitride with achieving technical result concerning reduction of content in them of inadmissible impurities and reduction of cost price of the materials produced.

As an information revealing an essence of the invention, it is necessary to note that the achievable technical result is provided with the help of the offered method of production of the metal silicon solution, which consists in that fact that the separation of silicon out of siliceous compounds is carried out in rector mainly out of oxides with the use of fluoro-halogen with a weight content of C_1 fluorine and C_2 halogen fitting the requirement $0,7 \leq (C_1 + C_2) / C_1 \leq 2,2$. Out of the heat produced in amount of E_2 in reactor there is the heat removed in amount of E_1 under the condition $1 \leq (E_1 + E_2) / E_2 \leq 2$. Removal of heat produced in reactor is necessary in connection with exothermic character of the reactions, which are conducted with so considerable energy release that the compulsory cooling is needed. Heat removal within the specified limits provides optimal conditions for passing technological process. The silicon binded by fluoro-halogen is decomposed electrolytically onto silicon and fluoro-haloid compounds with receipt of silicon in the fluid low-melting metal cathode. Fluorine and halogen allocated on the anode are transported for separation of silicon in n phases, the number of which is chosen within limits 1-3 under the condition $1 \leq (C_5 + C_6) / C_6 \leq 6,2$ where C_5 - weight content of fluoro-halogen, C_6 – weight content of the siliceous compound. Such cyclic repeated application of fluorine and halogen carrier is necessary for its rational use. The attendant components allocated on electrolytic cell are removed compulsory for the further utilization.

The achievable technical result is provided also by the offered method of production of the metal silicon, which consists in re-crystallization of silicon by evaporating under the lowered pressure out of metal silicon solution received by the above-described method, which has a weight content of silicon C_7 and a weight content of metal C_8 under the condition $1 \leq (C_7 + C_8) / C_7 \leq 10$. This is done while delivering to each unit of volume of the power solution in amount of E_3-E_4 under the condition $1 \leq (E_3 + E_4) / E_4 < 2$ and change of pressure over solution so to provide the condition $1 \leq (P_1 + P_2) / P_2 \leq 2$ where P_1 – fractional pressure of the solvent steams, P_2 – pressure of the saturated solvent steams at the atmospheric pressure.

The achievable technical result is provided also in the offered metal silicon received by the above-described method and characterized by content of impurities in it, dispersity and specific surface of its polycrystalline conglomerates. At that the especially pure material is received including silicon with a its weight content C_{11}, metal impurities with a weght content C_9 and non-metallic impurities with their weight content C_{10} under the condition $1 \leq (C_9 + C_{10} + C_{11}) / C_{11} \leq 1,1$ at a ratio of the minimal r_1 and maximal r_2

sizes of polycrystalline conglomerates, which fit the requirement $(1 + 10^{-5}) \leq (r_1 + r_2) / r_2 \leq 2$, and at a ratio of the minimal S_1 and maximal S_2 values of the specific surfaces of silicon polymetallic conglomerates fitting the requirement $(1 + 10^{-4}) \leq (S_1 + S_2) / S_2 \leq 2$.

The achievable technical result is provided also using the offered method of production of the ceramic materials on the base of silicon carbides and/or nitrides consisting in thermochemical interaction of the above-described metal silicon with nitrogenous and/or carbonaceous substance with a weight content C_{12} of carbon and/or nitrogen in substance, which has a weight content C_{13} under the condition $1 \leq (C_{12} + C_{13}) / C_{13} \leq 2$. At that interaction is carried out at a weight content of silicon C_{14} and a weight content of carbon and/or nitrogen C_{15} fitting the requirement $1 \leq (C_{14} + C_{15}) / C_{14} \leq 8$ till a weight content C16 of the free silicon in the finished ceramic material with a weght content C_{17} under the condition $1 \leq (C_{16} + C_{17}) / C_{17} \leq 1{,}8$ is achieved.

The achievable technical result is provided also in the ceramic material received by the above-described method, which is characterized by content of impurities in it, dispersity and the specific surface of its polycrystalline conglomerates, as well as by a content of its phase composition. At that the ceramic material is received, which includes metal impurities with their weight content C_{18}, non-metallic impurities with their weight content C_{19} and has a weight content C_{20} fitting the requirement $1 \leq (C_{18} + C_{19} + C_{20}) / C_{20} \leq 3{,}4$ at a ratio of the minimal r_3 and maximal r_4 sizes of polycrystalline conglomerates fitting the requirement $(1 + 10^{-6}) \leq (r_3 + r_4) / r_4 \leq 2$, and at a ratio of the minimal S_3 and maximal S_4 values of the specific surfaces of ceramic conglomerates fitting the condition $(1 + 10^{-6}) \leq (S_3 + S_4) / S_4 \leq 2$. The phase composition of the produced ceramic material, which is characterized by a weight content C_{21} of carbide and/or nitride □-phase and C_{22} of carbide and/or nitride□-phase, fits the requirement $1{,}05 \leq (C_{21} + C_{22}) / C_{21} \leq 1{,}9$.

It is necessary to note that the offered methods and materials have the same purpose, provide reaching the same technical result and thus are interconnected by the uniform inventor's idea described by the invention formula. Thus the concept of legal protection is based on the fact that continuity and coherence of the offered objects, as well as the supposed alternativeness of realization of the separate essential attributes or their combinations, predetermine non-traditional character of formulations of some attributes, for example, reflection of the features of the offered materials by characterizing not only the components contained, but also, in particular, characterizing the conglomerates and other parameters formed in them, which unequivocally characterize the materials received for practical realization of the task in view. These substances are received by realizing the technologically united set of methods interconnected by the uniform investor's idea.

To explain this, it is expedient to give the following example of practical realization of the offered method of production of the metal silicon solution. The silicon out of silicon dioxide or quartz sand is binded by fluorine- of halogen-carrier on the base of trifluorochlorine (ClF_3) under the reaction:

$$2SiO_2 + nClF_3 \xleftrightarrow{250^0\,C} SiF_4 + SiCl_4 + O_2.$$

SiF_4 and $SiCl_4$ formed in electrolysis are exposed to electrolysis by the method (I.V. Grankov Production of Silicon Abroad. /Обозинформ. Issue 1, - M., ЦНИИцветмет, 1983, p. 38; G. Mamontov and T.M. Laher Production of Fluorine by the Electrolysis of Calcium Fluoride – Containing Tetrafluoroborate Melts. //J. Electrochem. Soc. 1989, vol. 136, № 3, pp. 673-676), and the silicon solution of 22% mas. in zinc melt is received and on anode - the mix of fluorine and chlorine, which is used as a carrier for binding silicon out of silicon dioxide or quartz sand. Thus the most high-quality silicon solution is received at its low cost price. The optimal features of realization of operations of the method on production of metal silicon out of solutions consist in that fact that separation of silicon out of siliceous compounds is carried out mainly out of oxides using fluoro-halogen with a weight content C_1 of fluorine and C_2 of halogen fitting the requirement $(C_1 + C_2) / C_1 = 1{,}2$, and out of the heat produced in amount E_2 in the reactor a heat is removed in amount of E_1 under the condition $(E_1 + E_2) / E_2 = 1{,}4$. The silicon binded in such a way with the use of fluoro-halogen is decomposed electrolytically onto silicon and fluoro-haloid compounds producing silicon in the fluid low-melted metal cathode, fluorine and halogen allocated on the anode are transported for silicon separation in 2 stages $(C_5 + C_6) / C_6 = 2{,}6$ where C_5 – weight content of fluoro-halogen, C_6 – weight content of siliceous compound.

During practical realization of the offered method of production of the metal silicon, which consists in silicon re-crystallization by evaporating under the lowered pressure out of the metal silicon solution received under the above-described method, as a raw material the metal silicon solution is used with its high content C_7 not less than 15% mas. in the melt of metal. For example, a weight content of silicon C_7 and a weight content of metal C_8 under the condition $(C_7 + C_8) / C_7 = 3,7$ is chosen. The solution is evaporated bringing to each volume unit of the solution the amount of energy, the minimal E_3 and maximal E_4 values of which are kept under the condition $(E_3 + E_4) / E_4 = 1,6$, and changing the pressure over the solution in such a way to provide, in particular, the condition $(P_1 + P_2) / P_2 = 1,7$ where P_1 – fractional pressure of the solvent steams, P_2 – pressure of the saturated solvent steams at the atmospheric pressure. As an example of realization of this method with the absolute values of the parameters being kept, it is expedient to describe the following one. The solution of silicon of 22% mas. in the melted zinc is exposed to vacuum separation at 400^0 C and the temperature 400^0-450^0 C. The residual content of zinc in silicon powder at termination of separation under the pressure 10^{-4} mm hg makes < 1 ppm. While re-crystallizing silicon out of the melted zinc, the silicon of essentially higher quality than that by the known methods is received in kind of gray, hard, but rather fragile crystals of the specific weight 2,4.

Choice of the specified parameters of the technological methods within the declared limits allows producing the high-quality metal silicon, which is characterized by the content in it of the minimal amount of impurities, dispersity and the specific surface of its polycrystalline conglomerates within the permissible limits. As a result the especially pure material is received including silicon with its weight content C_{11}, metal impurities with a weight content C_9 and non-metallic impurities with their weight content C_{10} under the condition $(C_9 + C_{10} + C_{11}) / C_{11} = 1,0001$ at a ratio of the minimal r_1 and maximal r_2 sizes of the polycrystalline conglomerates, which fit the requirement $(r_1 + r_2) / r_2 = 1,7$, and at a ratio of the minimal S_1 and maximal S_2 values of the specific surfaces of the polymetallic silicon conglomerates meeting the requirement $(S_1 + S2) / S_2 = 1,6$. In particular, the received metal silicon with the total concentration of impurities 2×10^{17} at.cm^3 contains aluminium – 1×10^{16} at.cm^3, phosphorus and boron – $1,0 \times 10^{14}$ at.cm^3 at its low cost.

In case of practical use of the offered method of production of the ceramic materials on the base of silicon carbides and/or nitrides, which consists in thermochemical interaction of the above-described metal silicon with the nitrogenous and /or carbonaceous substance, often the values of the specified parameters are chosen as follows. The nitrogenous and/or carbonaceous substance is chosen with its weight content C_{13} at a weight content C_{12} of carbon and/or nitrogen in the substance chosen from the requirement $(C_{12} + C_{13}) / C_{13} = 1,5$. Interaction is carried out at a weight content of silicon C_{14} and a weight content of carbon and/or nitrogen C_{15} providing the condition $(C_{14} + C_{15}) / C_{14} = 2,8$ till a weight content C_{16} of the free silicon in the finished ceramic material with a weight content C_{17} is reached under the condition $(C_{16} + C_{17}) / C_{17} = 1,7$. Thus more quality and economic synthesis of ceramic materials by the described method is carried out, for example, keeping up the following absolute values of the parameters at interaction of the silicon received electrolytically with the purified carbonaceous and/or nitrogenous substances by the reactions:

$$Si + CN_4 = SiC + 2N_2$$
$$3Si + 4NH_3 = Si_3N_4 + 6H_2.$$

As a result the ceramic material is received by the above-described method, which is characterized by content of impurities in it, dispersity and the specific surface of its polycrystalline conglomerates, as well as the content of its phase composition. The ceramic material with high weight content C_{20} is received, for example, at availability in it of metal impurities with their weight content C_{18} and non-metallic impurities with their weight content C_{19} observing the requirement $(C_{18} + C_{19} + C_{20}) / C_{20} = 1,1$ at a ratio of the minimal r_3 and maximal r_4 sizes of polycrystalline conglomerates, which meet the requirement $(r_3 + r_4) / r_4 = 1,3$, and at a ratio of the minimal S_3 and maximal S_4 values of the specific surfaces of the ceramic material, which meet the requirement $(S_3 + S_4) / S_4 = 1,4$. The phase composition of the produced ceramic material, which is characterized by a weight content C_{21} of carbide and/or nitride □p̃hase and C_{22} of carbide and/or nitride □p̃hase, meets at that the requirement $(C_{21} + C_{22}) / C_{21} = 1,5$. As an example, it is expedient also to describe the received ceramic material by comparing the properties of silicon nitride and carbide (see the table).

The industrial application of the declared objects is proved by their wide use commercially, as well as by absence in the declared claims of any attributes difficult for practical realization.

The achievable technical result, as the data of experiments have showed, could be realized only by the interdependent plurality of all significant characters of the declared objects reflected in the invention's formula. The differences specified in it give a foundation for making conclusion about the novelty of such technical decision, and a plurality of the asked claims in connection with their unevidence – about its inventor's level, which is proved also by the above-stated detailed description of the objects declared. The declared significant distinctive characters, their lower and upper marginal values and those of the given analytical ratios were received on the base of statistical treatment of the results of experimental researches, analyzing and generalizing them and the known ones from the published data sources interconnected by the requirements on achievement of the technical result specified in the application, as well as with the use the inventor's intuition.

Thus the offered method of production of the polycrystalline silicon and its compounds – carbide and nitride – out of the natural siliceous concentrates allows producing the high-quality metal silicon and ceramic material in kind of silicon nitride and/or carbide with the minimal content of impurities and providing reduction of their cost price not less than in 1,5 times.

THE FORMULA OF THE INVENTION

1. The method of production of the metal silicon solution, which consists in that fact that separation of silicon out of the siliceous compounds is carried out predominantly out of oxides using fluoro-halogen with a weight content C_1 of fluorine and C_2 of halogen meeting the requirement $0,7 <= (C_1 + C_2) / C_1 <= 2,2$; from the heat generated in amount E_2 in the reactor the heat is removed in amount E_1 under the condition $1 <= (E_1 + E_2) / E_2 <= 2$; the silicon binded with the use of fluoro-halogen is decomposed electrolytically onto silicon and fluoro-haloid compounds receiving silicon in the fluid low-melting cathode, the allocated on the anode fluorine and halogen are transported for separation of silicon in n stages, the number of which is chosen within limits 1-3 under the condition $1 <= (C_5 + C_6) / C_6 <= 6,2$, where C_5 – weight content of fluoro-halogen, C_6 – weight content of the siliceous compound.

2. The method of production of the metal silicon consisting in re-crystallization of silicon by evaporating at the lowered pressure out of the metal silicon solution received according to the item 1, having a weight content of silicon C_7 and a weight content of metal C_8 under the condition $1 <= (C_7 + C_8) / C_7 <= 10$, delivering to each unit of volume of the power solution in amount E_3-E_4 under the condition $1 <= (E_3 + E_4) / E_4 <= 2$ and changing pressure over the solution in such a way to provide the condition $1 <= (P_1 + P_2) / P_2 <= 2$, where P_1 – fractional pressure of the solvent steams, P_2 – pressure of the saturated solvent steams at the atmospheric pressure.

3. The metal silicon produced according to the item 2, which includes silicon with its weight content C_{11}, metal impurities with their weight content C_9 and non-metallic impurities with their weight content C_{10} under the condition $1 <= (C_9 + C_{10} + C_{11}) / C_{11} <= 1,1$ at a ratio of the minimal r_1 and maximal r_2 sizes of polycrystalline conglomerates fitting the requirement $(1 + 10^{-5}) <= (r_1 + r_2) / r_2 <= 2$ and at a ratio of the minimal S_1 and maximal S_2 values of the specific surfaces of polymetallic silicon conglomerates fitting the requirement $(1 + 10^{-4}) <= (S_1 + S_2) <= 2$.

4. The method of production of the ceramic materials on the base of the silicon carbides and/or nitrides, which consists in thermochemical interaction of the metal silicon according to the item 3 with the nitrogenous and/or carbonaceous substance with a weight content C_{12} of carbon and/or nitrogen in the substance having a weight content C_{13} under the condition $1 <= (C_{12} + C_{13}) C_{13} <= 2$, interaction is carried out at a weight content of silicon C_{14} and a weight content of carbon and/or nitrogen C_{15} providing the condition $1 <= (C_{14} + C_{15}) C_{14} <= 8$ until a weight content C_{16} of the free silicon is reached in the finished ceramic material with a weight content C_{17} under the condition $1 <= (C_{16} + C_{17}) / C_{17} <= 1,8$.

5. The ceramic material received according to the item 4, which includes metal impurities with their weight content C_{18}, non-metallic impurities with their weight content C_{19} and has a weight content C_{20} while observing the requirement $1 <= (C_{18} + C_{19} + C_{20}) / C_{20} <= 3,4$ at a ratio of the minimal r_3 and maximal r_4 sizes of polycrystalline conglomerates fitting the requirement $(1 + 10^{-6}) <= (r_3 + r_4) / r_4 <= 2$ and at a ratio of the minimal S_3 and maximal S_4 values of the specific surfaces of conglomerates of the ceramic material fitting the requirement $(1 + 10^{-6}) <= (S_3 + S_4) / S_4 < 2$, phase composition of the ceramic material, which is characterized by a weight content C_{21} of the carbide and/or nitride □phase and C_{22} of the carbide and/or nitride □phase, fits the requirement $1,05 <= (C_{21} + C_{22}) / C_{21} <= 1,9$.

Table 1.

№	Ceramic material	Content of impurities, % of mas.		Phase composition, % of mas.		Average particle size, micron	Specific surface, m^1/g	Cost, $/kg
		metal	non-metallic	□	□			
1.	Si_3N_4	0,005	1,20	97,0	3,0	0,06	21-24	
2.	SiC	0,005	1,40	96,0	4,0	0,08	20-25	