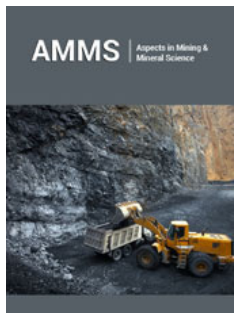


Influence of Technological Modes of Obtaining of Silicon Wafers on their Electro Physical Properties

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Abstract

In work process of pressing of powder silicon is considered. By experimental way dependence of relative density on pressure of pressing a fine-grained powder and a coarse-grained powder are defined. Temperature dependence of consolidation at sintering of powders Ni, WC, ZnC, AlNi and Si is shown. Dependence of specific resistance of the poly-Si wafers and factor of self-diffusion on heat treatment temperature. Quantitative results of the analysis of a chemical compound of a surface of grains and micro pictures of two kinds of the poly-Si are resulted.

Keywords: Powder silicon; Polycrystalline; Pressing; Solar cells; Sintering; Substrate

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Introduction

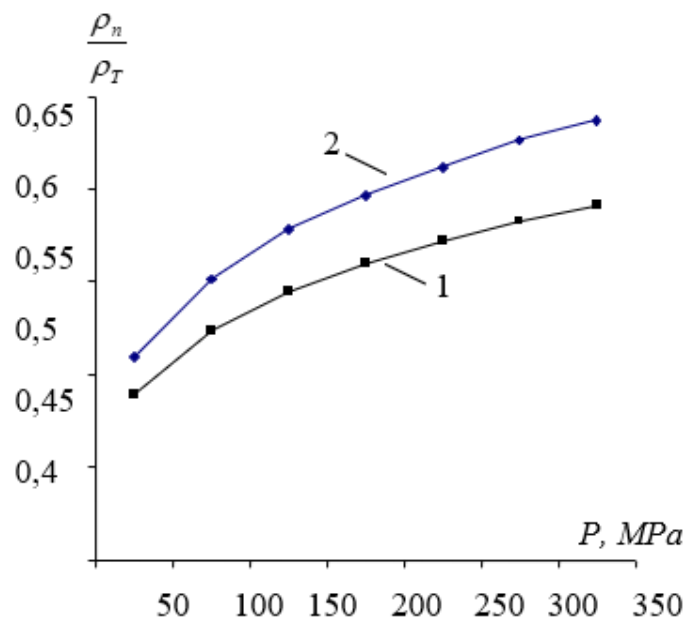


Figure 1: Dependence of relative density on pressure of pressing: a 1 - fine-grained powder and a 2 - coarse-grained powder.

Now exists much enough in the practical relation of the important materials with metal or quiz metal and co-valence or ionic and molecular character of the communication which obtaining are still insufficiently studied within the limits of powder technology [1,2]. One of such materials is the polycrystalline silicon applied as a perspective material for manufacturing of wafers for solar cells [3-5]. The urgency of carrying out of research in the given direction is

caused by possibility of obtaining without attraction of machining of thin ready preparations with the set sizes the maintenance of impurity at level $10^{-4} \div 10^{-6} \%$ and the size of grains $100 \div 1000 \mu\text{m}$ with application as substrates at sintering of quartz and sliced pyre graphite especially pure grades or special ceramic materials with sublimation temperature above 3000K . In the given work attempt to study the condition of consolidation of powders of silicon in a mode of hydrostatic pressing and solid phase sintering is undertaken. Powders of silicon with the average size of fractions $5 \div 10$ and $40 \div 60 \mu\text{m}$ obtained by crushing in a spherical mill and divided in the sizes on fibro cite placed $10 \mu\text{m}$ in elastic covers and then pressed hydrostatic pressure to 350MPa . The pressings obtained thus had relative density $0.6 \div 0.65$ and satisfactory durability. Compaction of powders of silicon with the small and large size of particles is shown on Figure 1.

The density of pressings increases with growth of pressure of hydrostatic pressing thus compaction of a fine-grained powder is less than coarse-grained. Such nonlinear dependence in a general view can be described in the form of sedate function:

$$(\rho_n / \rho_T) = ((\rho_n / \rho_T)_0 \times (P / P_0))^{1/m} \quad (1)$$

where (ρ_n / ρ_T) , P_0 - defined by practical consideration initial values of relative density and pressure m - an exponent characterizing an internal friction of powder weight (for a coarse-grained powder $m=8.7$ and for a fine-grained powder $m=10.3$). The

constancy of factor m in a wide interval of pressure testifies, Heat Treatment (HT) consolidation of a powder of silicon occurs only at the expense of inter partial sliding and turn of not deformable parts i.e. without chop off and crushing of particles.

Sintering of the formed preparations spent in vacuum at 10^{-5}Pa and to temperature $1120 \div 1650\text{K}$ within 30min the dependence of relative density. Obtained in the course of experiences on temperature for two samples with various granularity is resulted on Figure 2. Data resulted on Figure 2 show that HT process of sintering of powder preparations depending on temperature can be divided into two basic stages with the various mechanism of consolidation. At the first stage corresponding to a temperature interval to 1390K ($T_{\text{ann}}/T_m=0.82$) there is a slowed down consolidation under the influence of mainly one mechanism namely the mechanism of sintering of particles surface diffusion. At sintering temperature above 1440K ($T_{\text{ann}}/T_m=0.85$) there comes the second active stage of consolidation - transition of powder weight from a no equilibrium porous status with the developed free surface in equilibrium high density a status. The further grain in temperature to 1610K ($T_{\text{ann}}/T_m=0.95$) leads to the accelerated repacking of particles. On the basis of modern representations, it is possible to talk about process of sintering of substances HT in this area there is an intensive volume plastic current re-crystallization growth of grains (transformation of small angular borders to large angular) and formation of an equilibrium configuration of final polycrystalline structure [6,7].

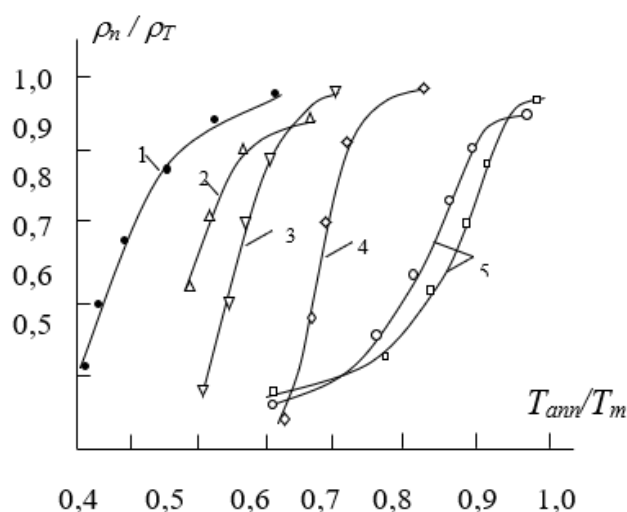


Figure 2: Temperature dependence of consolidation at sintering of powders: 1-Ni, 2-WC, 3-ZnC, 4-AlNi, 5-Si.

So high in comparison of powders of metals and other studied substances in communication by absence in the literature of direct correct data on the mechanism of this phenomenon it is possible to explain value of relative temperature of active sintering of a powder of silicon on visible the insignificant contribution of volume energy of dislocations in particles, and practically dominating contribution GB to energy i.e. silicon sintering it is carried out under action GB diffusions. For presentation of comparison of caking of a silicon powder on Figure 2 characteristic curves of growth of relative density are resulted at sintering of powders Ni, WC, ZnC, and AlN [8].

The arrangement of curves of dependence of relative density from the resulted temperature shows HT powders of metals and quiz metal connections actively conglomerate at temperature $(0.45 \div 0.6) T_m$ and definitively form the polycrystalline structure to $(0.7 \div 0.9) T_m$ and nitride of aluminum with is ionic-co-valence character of communication occupies intermediate position. It is characteristic HT at comparison of temperature dependence of factors of self-diffusion (Figure 3) active consolidation of all substances occurs in conditions when speed of volume diffusion are approximately identical i.e. $\lg D = 10^{-11} \div 10^{-14} \text{cm}^2/\text{s}$.

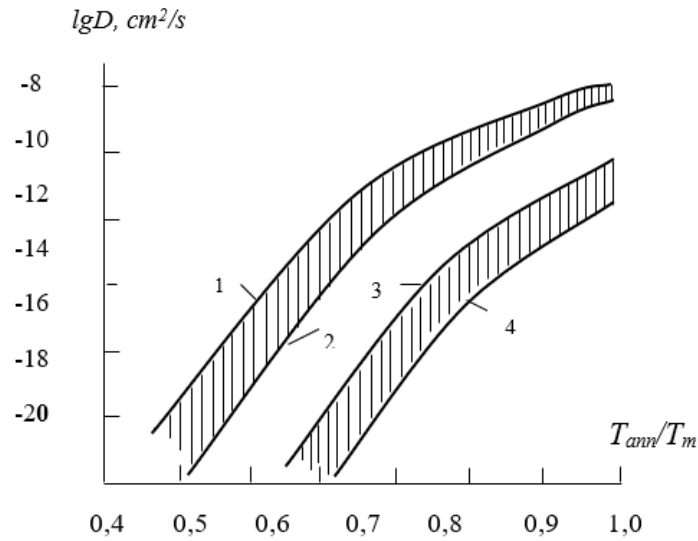


Figure 3: Temperature dependence of factor of self-diffusion: , , ,

This qualitative comparison once again proves HT rather the heat of sintering of silicon is connected as with low diffusion mobility and difficulty dislocation currents. Thus, resulted in the present work of data show about specificity kinetic formations of the poly-Si from powder weight and an urgency of search of new ways of the made active sintering at temperatures much more below temperature of fusion with a condition of obtaining of poly crystals with the reproduced properties close to properties of the poly-Si witch made on traditional technology.

Result and Discussion

Measurements of surface specific resistance of wafers depending on temperature and duration vacuum HT are spent.

Results are presented on Figure 4 (dependence of specific resistance on temperature HT) and on Figure 5 (dependence of specific resistance on duration HT). Apparently from the schedule (Figure 4) is available non monotonic dependence $\rho(T_{ann})$. Dependence $\rho(T_{ann})$ consists of three conditional sites: 1- $T_{ann} \leq 1200$ °C correspond to process of integration of grains; 2- 1200 °C $\leq T_{ann} \leq 1300$ °C to process of active diffusion of impurity; 3- 1300 °C $\leq T_{ann}$ to process of approach of uniformity. On 1 site semiconductor or metal dependence of conductivity on temperature is shown not. The qualitative kind of sites 2 and 3 corresponds to semiconductor conductivity. HT at $T=1200$ °C leads a material to a semiconductor status.

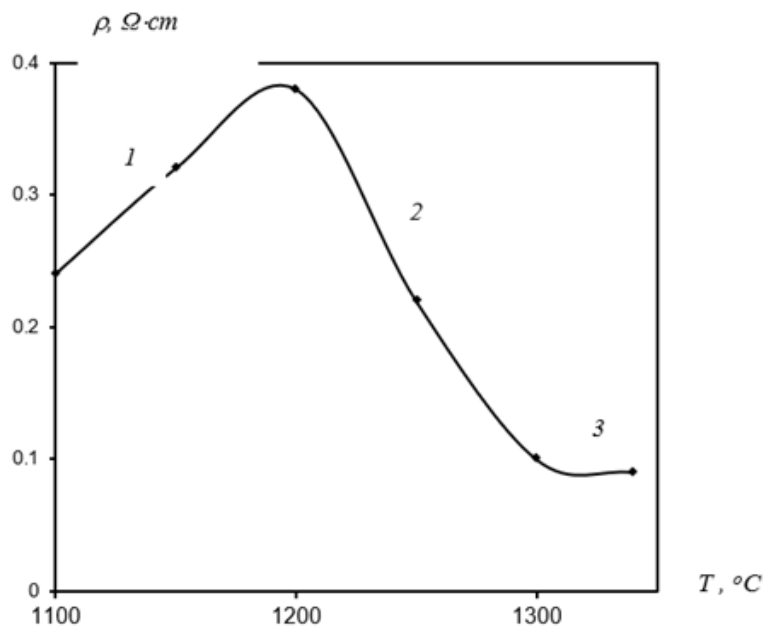


Figure 4: Dependence of specific resistance of the poly-Si of wafers on temperature HT.

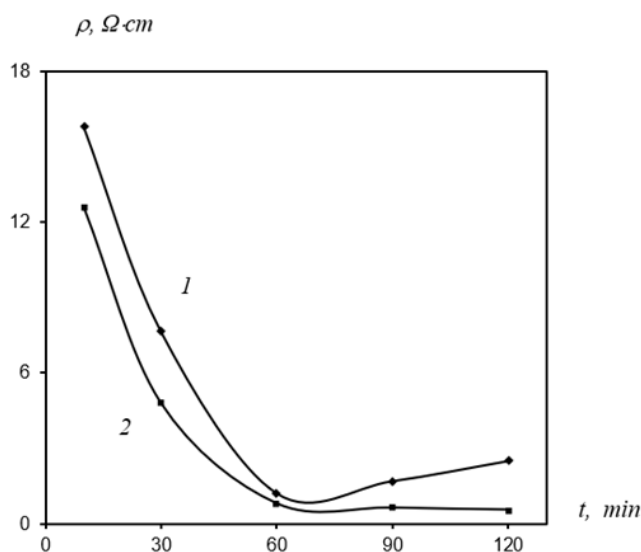


Figure 5: Dependence of specific resistance of the poly-Si wafers on duration of HT at T=1200 °C; 1-d=1mm; 2-d=2.5mm.

Dependence ρ from duration (Figure 5) shows HT optimum duration (Δt) at $T_{ann}=1200$ °C makes HT not less than 60 minutes. Naturally $\rho(\Delta t)$ depends and on a thickness of wafers. With reduction of a thickness of value ρ increases and at $\Delta t > 60$ min monotony of dependence $\rho(\Delta t)$ is broken HT on visible connected with pauperization of grains volume by the basic charge carriers.

From this schedule it is visible HT the qualitative course of curves does not depend on the thickness of a plate volume HT hence is provided a semiconductor material. Dependence ρ from duration HT at T=1250 °C (Figure 6) testifies to a deviation of parameters of a wafer from optimum modes HT (duration and temperature).

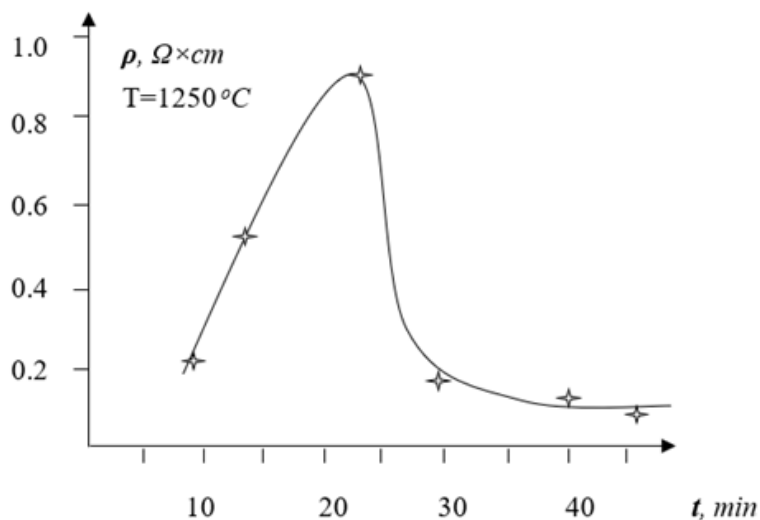


Figure 6: Dependence of specific resistance of the poly-Si wafers at duration HT at T=1250 °C.

Measurement of lifetime of charge carriers in the wafers obtained by HT at T=1200 °C and duration $\Delta t=20$ min testified to suitability of the given material for manufacturing effective SC. As a result of the analysis of dependence on temperature HT is shown lifetime of charge carriers satisfactory enough consent with data of sound measurements of surface specific resistance. For finding-out of the mechanism of change of quality of a material at HT experiments on detailed studying of dependence of specific

resistance of a wafer and also concentration of the basic charge carriers and their mobility four sound are made by a method. Apparently from the schedule (Figure 7) as a result HT at T=1200 °C and duration $\Delta t \leq 70$ min are observed a semiconductor course of dependence $\rho(\Delta t)$. At $\Delta t \geq 70$ min occur some growth ρ (a curve 1) connected apparently to diffusion of various impurity or with saturation of process of integration of grains. Some samples have a maximum at duration of 90 min and further decrease ρ .

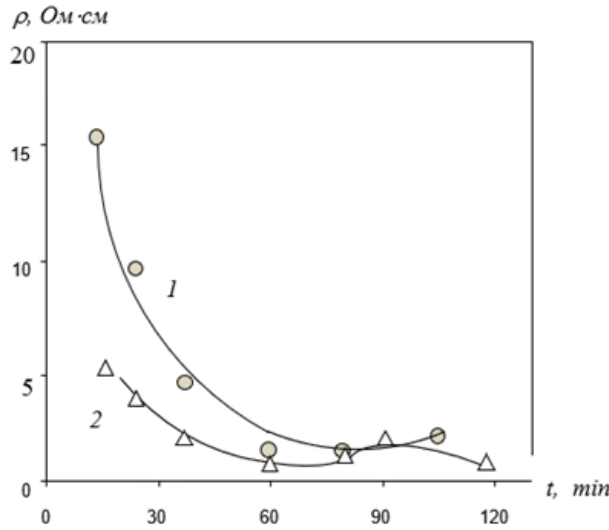


Figure 7: Dependence of specific resistance of the poly-Si wafers with various reference values from duration HT.

At increase T to 1250 °C time of transition from metal on the semiconductor is reduced (Figure 6) the transition point corresponds $\Delta t=25\text{min}$ (for $T=1200\text{ °C}$ and $\Delta t=60\text{min}$). It is necessary to notice HT at $T=1250\text{ °C}$ value ρ grows in 4 times when at $T=1200\text{ °C}$ growth ρ made all in 2 times. At $T=1250\text{ °C}$ ρ has decreased in 4 times for 10min when at $T=1200\text{ °C}$ ρ has decreased in 4 times for 30min. In other words, transition to a semiconductor status (qualitatively) occurs almost twice as fast at $T=1250\text{ °C}$ than $T=1200\text{ °C}$. Dependence of concentration of charge carriers on modes HT (Figure 8) in particular $n(\Delta t)$ is defined and established a maximum at $\Delta t=90\text{min}$ ($T=1200\text{ °C}$). At this mode optimum (homogeneous) activation of an alloying

impurity is reached. At longer HT starts to play a role diffusion of impurity to GB hence decrease in concentration of charge carriers. By calculation dependence $\mu(\Delta t)$ for the samples subjected HT is defined at $T=1200\text{ °C}$ (Figure 9). With growth of concentration of a charge carriers decreases μ and the minimum is reached at $\Delta t=90\text{min}$. Further decrease in concentration value μ in direct ratio to a thickness of a wafer HT testifies to existence of grains with the sizes comparable with thickness of a wafer leads to some growth of μ . Wafers from the cast poly-Si showed non-uniform changes ρ with growth of temperature [1]. Such research allow to open physical properties of a material on the one hand and to expand functional area of its application - with another.

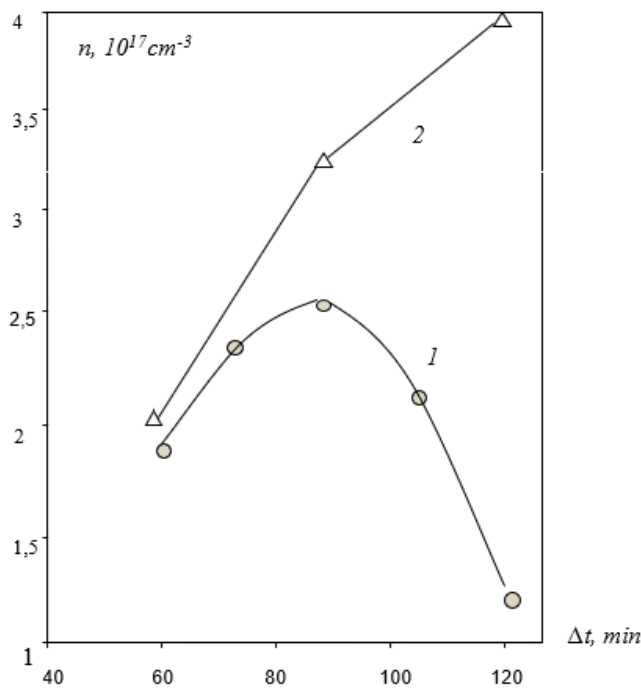


Figure 8: Concentration dependence of charge carriers on HT duration.

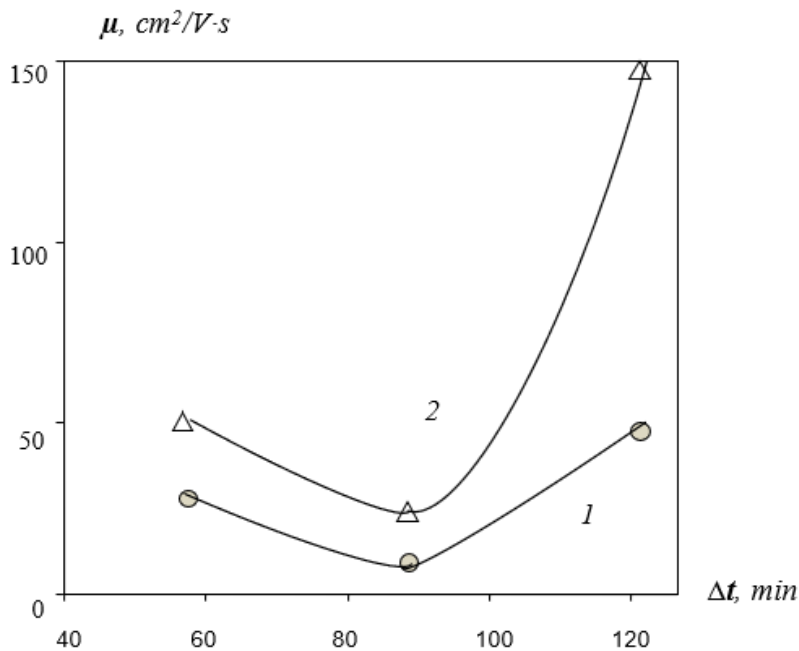


Figure 9: Dependence of mobility of charge carriers on duration HT.

Therefore, similar research of the poly-Si wafers obtained from powder raw materials are carried out. On Figure 10 & 11 curve dependences of specific resistance of the poly-Si wafers obtained HT are resulted at T=1200 °C and Δt=30min (1) also Δt=60min and Δt=90min (2) from an ambient temperature. From curves it is visible that with duration growth HT change ρ with temperature becomes stable. Non-uniform change ρ with temperature in range

T≤300 °C is replaced with stable growth at T≥400 °C. As a whole physical properties of studied wafers are similar to properties of wafers from the cast poly-Si. Thus, the basic electro-physical indicators of the silicon wafers made in the offered way correspond to parameters typical SC. Thus, the sizes of crystal grains reach values 80÷100μm characteristic poly-Si for the high-quality films used for creation thin-film SC with high efficiency [8].

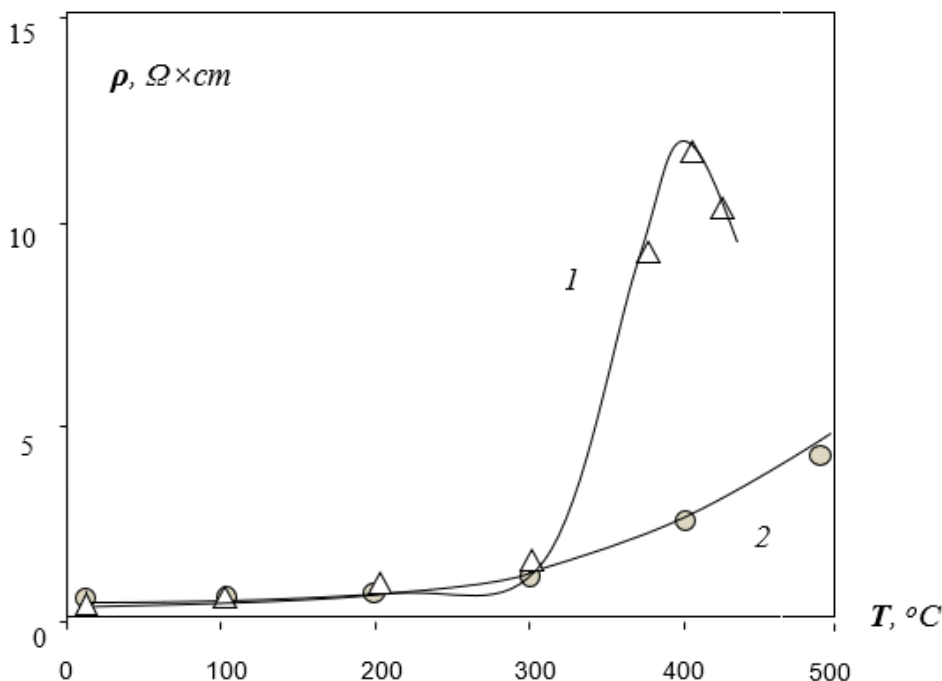


Figure 10: Dependence of specific resistance of the poly-Si wafers obtained HT at T=1200 °C and Δt=30min (1) and Δt=90 min (2) from temperature.

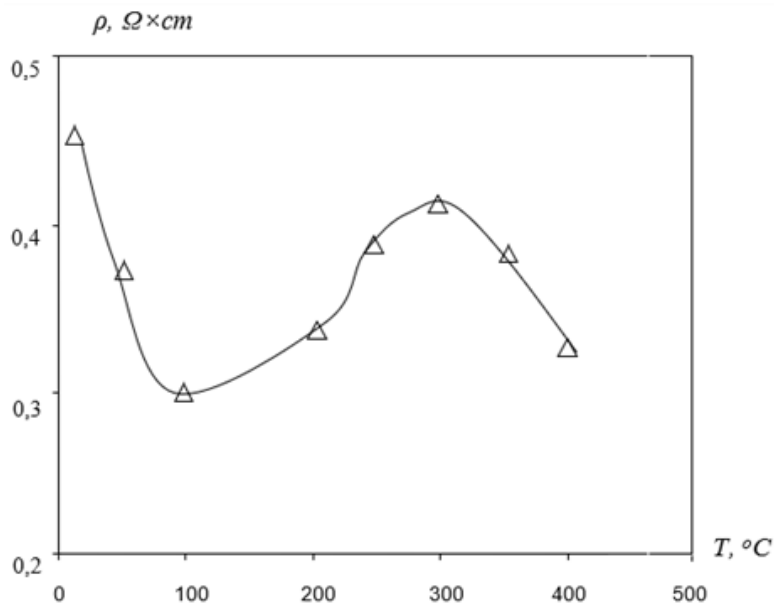


Figure 11: Dependence of specific resistance of the poly-Si wafers obtained HT at T=1200 °C and Δt=60min from temperature.

The choice of optimum modes of technology allowed to obtain of the poly-Si wafers in the thickness 250÷400μm and diameter 40÷125mm. Application of an offered way provides possibility of obtaining of wafers and larger sizes (more than 150mm) thus the form of made wafers can be both round and rectangular or difficult for example triangular or ring form HT allows to simplify manufacture and to lower losses at manufacturing SC intended for use in solar batteries. Microscopic research of the surface of the poly-Si wafers and the analysis of a chemical compound of a material both in the field of GB and in grains are carried out. The

spectrogram of the investigated of the poly-Si wafers is resulted on Figure 12. Quantitative results of the analysis of a chemical compound of a surface of grains and micro pictures of two kinds of the poly-Si are resulted in Table 1. As follows from the table, areas GB are sated by oxygen complexes. It is visible HT the surface of grains in all cases abounds with various difficult structures. We will notice HT grains with a size 100÷300μm possess a rough surface with the sizes of ledges and deepening with size to several μm. Also follows HT concentration of impurity increases from the center of grain to edge of its surface.

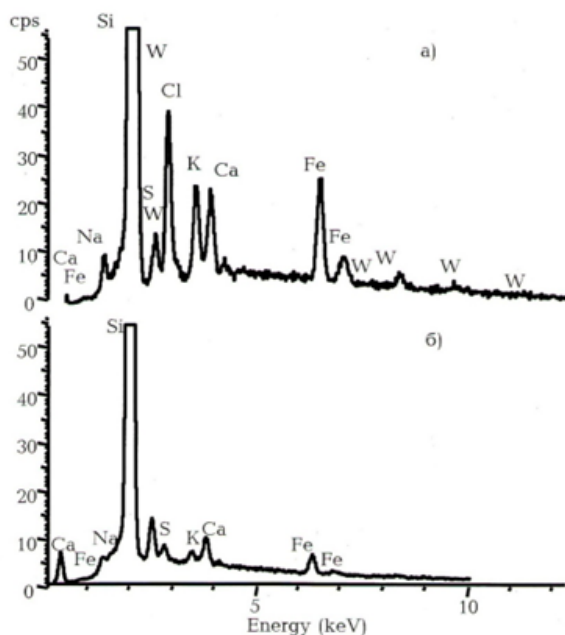
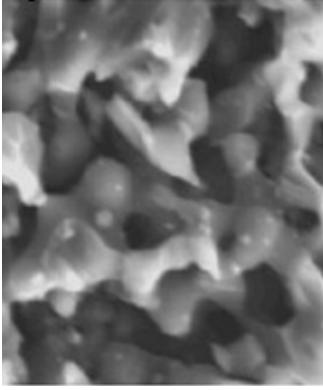
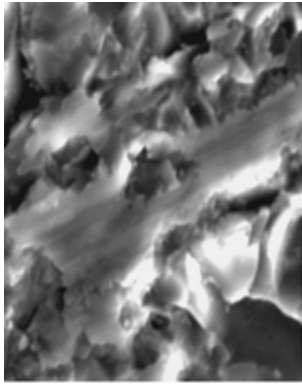


Figure 12: Spectra of distribution of impurity on a surface of the poly-Si wafers before (a) and after (b) two stage clearings.

Table 1: Results of scanning of a surface of polycrystalline silicon wafers obtained by various methods and impurity structure.

Element	The Polycrystalline Silicon Received from Powder Raw Materials		The Polycrystalline Silicon Received by Secondary Moulding	
	Element, %	Atom, %	Element, %	Atom, %
Si K	43.14	94.50	70.65	98.34
S K	0.80	1.54	1.08	0.52
Cl K	0.28	0.48	0.45	0.34
Na K	0.40	1.08	-	-
K K	0.05	0.08	-	-
Ca K	0.52	0.80	0.17	0.24
Fe K	1.38	1.52	0.23	0.21
Co K	-	-	-	0,12
Mg K	-	-	-	0,23
All:	46.58	100.00	72.58	100.00
Micrograph surface of poly-Si plate				

In the spent experiments have paid attention to cleanliness of an initial material (powder) the chamber and the furnace for HT. Separate experiments on additional magnetic clearing of a powder and vacuum clearing of the formed wafers are put. For clearing of a powder of silicon of extraneous impurity two ways are used. In the first way, grinding balls in a mill silicon pieces are used. In the second way procedure is used in two stage. The first stage consisted of transporting an initial powder through a magnetic field. Procedure of clearing of a powder by a magnetic field allowed to operate speed of transport of a powder and size of intensity of a magnetic field. Concrete modes of this procedure are saved in the form of a know-how since their patenting is provided. For clearing of poly-Si wafers surface from easy evaporation impurity the second stage - endurance is used at temperature of ~ 100 °C in the conditions of vacuum. The positive result of this way can be seen by comparison of the initial and cleared wafers surfaces (Figure 12). HT can have stimulated an effect and on processes of carrying over of a charge through barrier structures photosensitivity of a wafers hence to I-V and photoelectric characteristics of structures with the p-n-junction created on their basis.

Conclusion

Optimum modes of thermal stimulation of properties of the formed poly-Si of wafers and structures on its basis, in particular, integration of the sizes of grains, powder, solid-state re-crystallization, restriction defects, modulation of conductivity,

adhesion improvement, etc. are defined. On the basis of microscopic research of the wafers obtained at various technological modes the mechanism of integration of separate grains and growth of crystalline at temperatures below temperature of fusion silicon based on the account of density of save up GB i.e. is offered their energy of activation. The analysis impurity structure of a surface of the obtained wafers by using of technique "Cameca" is carried out and is shown HT fused enough sites of a plate consists of atoms Si ($99.16 \div 100\%$) and areas of borders of grains contains Si- $54.65 \div 97.56\%$; S- $0.88 \div 1.38\%$; Cl- $0.45 \div 0.64\%$, Ca- $0.17 \div 0.21\%$; Fe- $0.21 \div 0.23\%$.

The basic electro-physical parameters of the poly-Si wafers obtained at optimum modes of developed technology are defined: specific resistance $1 \div 10 \Omega \times \text{cm}$ and lifetime of non-basic charge carriers $10^{-7} \div 10^{-6} \text{s}$ (average the size of grains $10 \div 100 \mu\text{m}$ and a thickness of wafers $0.25 \div 0.5 \text{mm}$). Their suitability for use as a substrate for the epitaxial films ready wafers for SC and other devices is proved. The choice of optimum modes of technology allowed to obtain wafers in the thickness $250 \div 400 \mu\text{m}$ and diameter $40 \div 125 \text{mm}$. Application of an offered way provides possibility of obtaining of wafers and larger sizes (more than 150mm) thus the form of made wafers can be both round and rectangular or difficult for example triangular or ring form and HT allows to simplify manufacture and to lower losses at manufacturing SC intended for use in solar batteries.

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