Dependence of specific resistance for polycrystalline silicon plates obtaining based on powder technology to mold parameters

Tulkun Nasirov [1,*], Murod Nosirov [2], Sevaraxon Jonibekova and Valixon Abduazimov [2], Umidaxon Mirzayeva [3]

[1] University of geological sciences, 64, Olimlar str., Tashkent, Uzbekistan; tulkunnasirov@yandex.ru
[2] Andijan state university, 129, Universitet str., Andijan, Uzbekistan
[3] Andijan state technical institute, 56, Bobur shox str., Andijan, Uzbekistan

Abstract

The features of the production of polycrystalline silicon plates based on powder technology and the experimental results of dependence of the specific resistance for the obtained samples on the temperature, time and thickness of the samples are processed using the "least squares" method have been considered. The new expression that describes these dependencies has been obtained which allows to select optimally the remaining two values. When any of these values is constant, as well as to make a theoretical analysis of dependence of the specific resistance on the third value, then other two of these values are constant.

Keywords: polycrystalline silicon, powder technology, specific resistance, firing duration, firing temperature, the least squares method

1. Introduction

It is known that the polycrystalline (PC) silicon pieces is the material which consists many small silicon crystals. Because of unique features and small producing costs in comparison of the monocrystal silicon PC silicon is the important material in the semiconductors and solar energetics branches. Despite the reached advantages production of PC has several modern difficulties affecting to the technologic, ecologic, economic and safety aspects [1]. The many investigations on using in the solar energetics within the scientific society are being conducted. These investigations cover the broad area themes dealing with the development of producing technologies, efficiency of solar panels and growing their strength [2].

Production of PC plates using powder tecnologies is the complex technologic process which includes the several main stages. This method based on creating monolit plates from the silicon powder. Later these plates for producing the different semiconductor devices, including solar batteries can be used [3].

In paper [4] the experimental device which allows to study the model losses consisting Si disc resonator and situated around electrode in 100◎300oK temperatures range had been developed. Based on the dependence of specific resistance of Si on temperature the dependence of introduced by electric field of the mechanical losses on the Si specific resistance which is in the qualitative agreement with the theoretical model had been constructed. The obtained results allow to calculate the mechanical losses appearing because of electrostatic field's effect actuators to testing masses and corresponding noises.

Author of paper [5] had considered amorpha hydrogenated Si and its structure and electrophysical properties of films where influencing degree of laser radiation parameters to properties of films for amorpha hydrogenated Si had been analyzed. In paper [6] the complex investigation of technical carbon had been carried out: the roentgen structure's analysis and electronic microscopy had been studied; the specific resistance, oil absorption coefficient, surface area on multipoint nitrogen adsorption and external surface area on nitrogen had been measured. It had been shown that the dependence of specific resistance on the structure parameters is describing qualitatively by linear regression equations.

In paper [7] ZnO:Te/Si(111) polycrystal films by gas phase epitaxy in hydrogen method in the flowing reactor with a low pressure had been obtained. It had been shown that in the radiation spectrum of ZnO:Te/Si(111) films the total range of the visible part of spectrum is observed.

By authors of paper [8] the phase composition, structure, particles and agglomerates size constructing by powder annealing obtained from hydrophobized Si organic liquid in the presence of aluminum powder had been investigated. It had been found that the annealing temperature change (from 550 up to 700oC) influences to average size of individual particles and aluminum additive composition effects to the agglomerates size.

In paper [9] the radiation resilience of Si and SiC had been compared. It had been shown that unlike Si, the relatively small difference in the velocity removing charge carriers in SiC deals with that annealing the first radiation defects in the absorption process is absent practically. Author of paper [10] had investigated the transverse chips of powder Si in dependence of anode etching duration.

Authors of paper [11] had analyzed the temperature dependence of filiform Si crystals grown in an open system in the framework of growth of controlled by heterogeny chemical reaction on the liquid-gas boundary. In paper [12] the tensor properties of structures with Schottky barriers by impact of momentum hydrostatic pressure had been investigated. It had been shown that the dependence of relative variation of direct current by constant value of impact of hydrostatic pressure on the electric voltage deals with the presence of compensating impurities in the base material volume of investigating structures with Schottky barriers.

Therefore, development of PC silicon based on powder technology, controlling and improving the electrophysical parameters of obtained samples are one of the present's actual tasks. In the present paper the features of producing PC silicon based on the powder technology and dependence of the specific resistance on the firing temperature, firing time and sample's thichness are investigated.

For papers that report original research, you should use the titles "Materials and Methods", "Results", "Discussion" and "Conclusions" (optional).

2. Materials and Methods

The raw material in the powder technology maybe the technical silicon. It in the electric fire with carbonate through decreasing the silicon oxide is produced. The purified silicon to the small powder state is reduced. The powder into molds is poured and in order to creating samples of needing the shape and sizes under the high pressure is pressed. The pressed samples in the anti-oxidation environment at the high temperature are fired. It can be the vacuum or inert gas atmosphere. The powder particles at the firing process in order to the monolithic structure are connected [13].

As is known the firing process of silicon at high temperatures is realized. It depending on the features and using one can be reach up to 1000-1400oC. The maintenance time on the firing temperature is changing which can be varied duration from the several second up to hours. This duration depends on the volume, temperature and other criteria. After ending the firing process the material is being cooled. In order to preventing defects, the cooling temperature is controlled [14].

The main mechanism of the firing is atoms distribution and this powder leads to the grain's unification and decreasing defects [15]. Material is squeezed and grain's structure is created. In the firing process the main defect of material is decreasing which leads to increasing density and improving the electric features. Atoms are moving around of grain boundaries which helps the grain's growth and improve of the crystal structure. On dependence of heating processing the grain's growth in the PC silicon obtained by powder technology is the important aspect affecting to its physical end electrical features. This process consists usually the several main stages:

– in the initial heat processing duration in the initial stage creation of the initial grains is realizing and small grains begin grow because of atom's distribution in the material;

– the growing velocity of grains is increasing because of increasing the temperature in the growing stage; this circumstance deals with increasing the diffuse velocity of atoms around the grains boundaries and decreasing their activation energy; then the grains are growing and begin interact themselves;

– in the unification stage grains begin to come together in order to create bigger crystals and decreases quantity of grain boundaries; this circumstance improves the crystal structure and decreases porosity;

– grains in the stabilization stage reach to the final volume and growth will be insignificant; the stabilization process in the known conditions may consist creating the new phases and increasing volume.

The grain boundaries create barriers for movement of charge carriers. The firing temperature influences to the barrier's height and width. The barrier's height in optimal conditions is minimized which decreases of specific resistance. Distribution around grain boundaries of alloy elements influences to resistance too. Increasing the firing temperature helps to uniform distribution and decreasing concentration of mixtures. Increasing the firing temperature helps to uniform distribution concentration of mixtures which can decrease or increase the resistance in dependence of mixture's concentration and type.

In order to choose the optimal parameters of obtaining samples in processing the experiment results we use "the smallest quadrates" method [16]. It is known if the results obtained experimentally for two physical quantities (x_i, y_i) are linearly in the theoretical point of view that is expressed by law

$$y_n=A+Bx \quad (1)$$

then we must find such A and B coefficients, in result the theoretical and experimental obtained values for parameters y_i will closest to each other. For that the smallest quadrates errors must have a minimal value

$$\chi^2 = \frac{1}{N}\sum_{i=1}^{N}(y_i - y_i^n)^2 = \frac{1}{N}\sum_{i=1}^{N}(y_i - A - Bx_i)^2 \quad (2)$$

that is following conditions mast satisfy

$$\begin{cases} \partial\chi^2 / \partial A = 0, \\ \partial\chi^2 / \partial B = 0 \end{cases} \qquad (3)$$

If we to expression (3) put to representation (2) and solve created equations system then one obtained following expressions for define of A and B coefficients

$$\begin{cases} A = \dfrac{\sum y_i \sum x_i^2 - \sum x_i \sum x_i y_i}{N\sum x_i^2 - (\sum x_i)^2} \\ B = \dfrac{\sum y_i - NA}{\sum x_i} \end{cases} \qquad (4)$$

This method for arbitrary form of theoretical in according to connections between of experimental results can be generalized.

3. Results

Specific resistance of the PC silicon plates depends strongly on the duration of firing in given temperature. Increasing of the firing duration strengths the distribution of defects in the alloy compounds and crystal structure. It leads to more uniform distribution and can decrease the point like defects concentration. On the increasing the firing duration are take place the grain's growth and unification of small grains. It leads to decreasing the grain boundaries quantity which decreasing the resistance. Moreover, one helps to decreasing defects and increasing the material density which influences positively to its conductivity that is one allows to recombination and destroy some defects holding the charge carriers. In result, the specific resistance of samples is decreasing.

We had obtained the following results when one tested the dependence of specific resistance on the firing duration (See Table 1).

Table 1. Experimental results for the dependence of specific resistance on the firing duration

t, hours	0.17	0.33	0.50	0.67	0.83	1.0	117	1.33	1.50	1.67	1.83	2.0
ϱ, Ohm·cm	6	4	3	2	1.4	1.0	1.1	1.5	1.8	2.2	2	1

We assume dependence of the specific resistance on the firing duration as

$$\rho(t) = \rho_0 \sum_{i=0}^{n} a_i t^i \qquad (5)$$

and for defining values of unknown coifficients ρ_0 and ai use "the smallest quadrates" method. The initial calculations showed that this representation represents on the qualitative level the experiments results in case of *n*=3. The results obtained for this case are represented on the Figure 1 and Table 2.

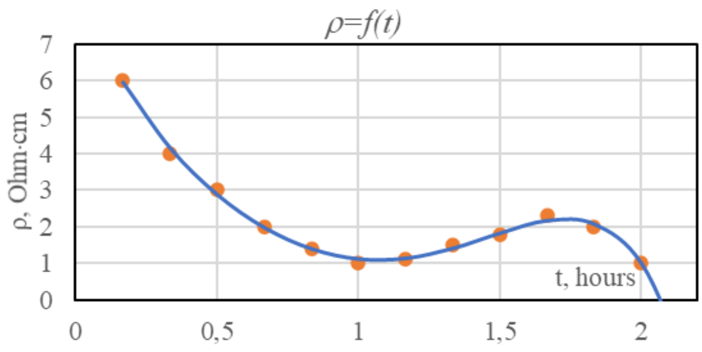


Figure 1. Dependence of specific resistance on the firing duration. Here dots correspond to experimental results and dashed line represents the theoretical calculated values.

As we see from Figure 1 that theoretical calculated results are too close with the experimental ones. This situation can be sight from the values obtained by "the smallest quadrates" method (χ^2=0.022) too (see Table 2).

Table 2. Values of ρ_0 and a_i coefficients obtained using "the smallest quadrates" method

n	a_0	a_1	a_2	a_3	χ^2
3	8.53	-18.58	21.3	-18.27	0.022

The thin plates have a high velocity of heat and compounds distribution which leads to the fast stable state in the heat processing. This can lead to decreasing the resistance in comparison of the thicker plates. The process reaching to a stable state can be happen slower in case of thicker plates, the resistance in case of long heat processing time is changing slowly.

In experiments carried out for testing the dependence of the specific resistance on the plate thickness we obtained the following results (Table 3).

Table 3. Values of specific resistance in the different diameters

d, mm	0.5	1.0	1.5	2.0	2.5
ϱ, Ohm·cm	2	1	2	4	5

The dependence of the specific resistance on the plate thickness also we assume as

$$\rho(d) = \rho_0 \sum_{i=0}^{n} a_i d^i \qquad (6)$$

and for defining unknown ϱ0, and ai coifficients one uses "the smallest quadrates" method. Initial calculation results for n=3 case are represented on Figure 2 and Table 4.

As we can see from the Figure 2 the theoretical calculated results too close with the experimental results and the smallest quadratic value (◎2=0.022) for n=3 case is the smallest one.

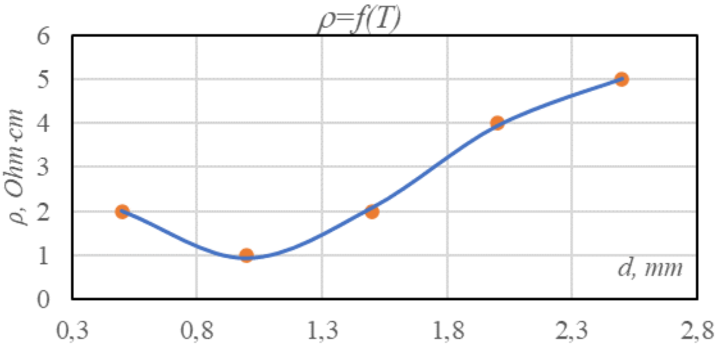


Figure 2. Dependence of specific resistance on the plates thickness. Here dots correspond to experimental results, dashed line represents the theoretical calculated values.

Table 4. Values of ρ_0 and a_i coefficients calculated using "the smallest quadrates" method

n	a_0	a_1	a_2	a_3	χ^2
3	6.8	-14.29	10.43	-2.0	0.023

At high firing temperature we reach to the high crystallization stage of material, the defects on structures are minimized and the monolithic structure having good conductivity is created. Moveability of charge carriers reaches to the maximal stage because of the grain boundaries are activated slowly in point of view retardation and distribution of carriers. Quantities of defects in the material structures and holes are decreased essentially which increases conductivity.

As when the firing temperature gets up then grain's volume of the PC is increased. The density of the bigger grain's boundaries is smaller which it decreases distribution of the charge carriers in these boundaries and increases their moveability. The high firing temperature helps to decrease vacancies holding charge carriers, decreasing their moveability and point like defects quantity as intermediate atoms. The grain boundaries can be barrier to movement of charge carriers. The high firing temperature helps to decrease the barriers height in the grain boundaries because of grain's recovery and growth.

The firing temperature defines connectedness strongly and effectively of the silicon grains. Moreover, it influences to quantity and type of the defects in the grain boundaries. As the firing temperature is growing the grain volume of the PC silicon is growing too. The density of grain boundaries for bigger grains is smaller which decreases distribution of charge carriers on boundaries and increases the moveability. The high firing temperature helps to decrease holding the charge carriers and decreasing their moveability vacancies and a pointing defects quantity as the intermediate atoms. The grain boundaries maybe as barriers to the charge carrier's motion. Because of the grain recovery and growth, the high temperature helps to decrease the barriers height at grain boundaries.

The firing temperature determines whether how tightly and effective it is connected themselves of the separate silicon grains, moreover it influenses to the defects quantity and type on the grain boundaries. As the firing temperature is growing the silicon grains are growing. On the dependence of growing the firing temperature the silicon grains are getting bigger. This situation decreases the grains boundaries quantity needing for crossing the charge carriers and influence of potential barriers on the grain boundaries. The high firing temperature helps to decrease defects as dislocations and holes in the silicon structures. It leades to distributing and decreasing recombination the charge carriers in the defects. At the high firing temperatures the active centers capable of delaying the grain boundaries and charge carriers will be less. Because of the defects and high density of holes the specific resistanse usually, ⊚ will be high. On the growing temperature parameter ⊚ is decreasing, on the optimal firing temperature reaches to the minimal point, after that starts to grow because of appearing new defects. In order to test the dependence of the specific resistance on the temperature we ontained the following results (see Table 5).

Table 5. The obtained results for the dependence of the specific resistance on the temperature

T, ^{0}C	1000	1050	1100	1150	1200	1250	1300	1350
ρ, Om·cm	11	8	5	3	1.8	1	1.2	2

The dependence of the specific resistance on the temperature we assume as following representation

$$\rho(T) = \rho_0 \sum_{i=0}^{n} a_i T^i \qquad (7)$$

and in order to define unknown ϱ0 and ai coifficients use "the smallest quadrates" method. The initial calculations showed that the experiment results in case of n=3 (3) are characterized quantitatively. The initial calculations results in case of n=3 on Figure 3 and on Table 6 have been represented.

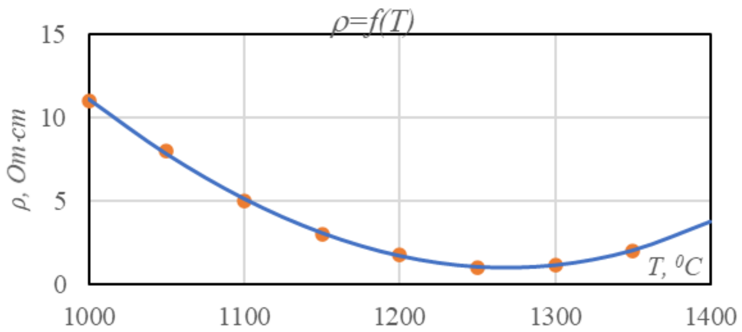


Figure 3. Dependence of specific resistance on the temperature. Here dots correspond to experiment results, dashed line represents the theoretical calculations.

Table 6. Values of ρ_0 and a_i coefficients calculated using "the smallest quadrates" method

n	a_0	a_1	a_2	a_3	χ^2
3	140.67	-0.135	$-4.7\cdot10^{-5}$	$-5.25\cdot10^{-8}$	0.035

As is seen from Figure 6 and Table 3 that the theoretical results calculated according to representation (7) are too close with the experimental values in case of n=3. Then value for the average quadratic error equals to χ^2=0.035.

It follows from results represented above that the specific resistance of the firing duration depends on the firing temperature and cub of the plate width. Therefore for the specific resistance we can write

$$\rho(y) = \rho_0 \sum_{i=0}^{3} a_i y^i = \rho_0 \sum_{i=0}^{3} a_i \left(\frac{tT}{d}\right)^i \qquad (8)$$

Here t is the firing duration (in hours), T is the firing temperature (in ^{o}C), d is the plate width (mkm).

From representation (8) for case of n=3 we rewrite as

$$\rho(y) = \rho_0(a_0 + a_1 y + a_2 y^2 + a_3 y^3) \qquad (9)$$

As the differential analysis of this representation on argument y from condition

$$\rho'(y) = 3a_3 y^2 + 2a_2 y + a_1 = 0$$

it follows in points

$$y_{1,2} = \frac{-a_2 \pm \sqrt{a_2{}^2 - 3a_1 a_3}}{3a_3} \qquad (10)$$

function ρ(y) in critical points, that are at y_1=0.964 point reaches minimum and y_2=1.635 point have the maximal values.

Thus, condition

$$0.964 = \frac{tT}{d} \qquad (11)$$

allows us to choose two parameters from t, T and d when arbitrary one them is constant. That is

when *T*=const then *t*/*d*=const;
when *t*=const then *T*/*d*=const;
when *d*=const then *tT*=const.

Moreover, representations (8)-(9) allow to analyze theoretically the dependence of the specific resistance on third parameter when arbitrary two parameters of t, T and d are constant.

The dependence of specific resistance on the firing temperature when plate width parameter d is const (0.8 mm, 1 mm, 1.2 mm) and on the different values of firing duration t is represented on Figure 4. It is seen from Figure 4a that when d=0.8 mm and t=0.8 and 1 hour then the specific resistance is proportional to the firing temperature, at the same time if t=1.2 and 1.4 hours then one is inversely proportional. As to the Figure 4b we can sight that when d=1 mm and t=0.8 hour then the specific resistance reaches the minimal value by T=1200 ^{o}C, at the same time in case of firing duration equals to 1-1.4 hours then one is increasing proportionally to the firing temperature. Finally, it is seen from Figure 4v that when d=1.2 mm and t=0.8 hour then the specific resistance is proportional to the firing temperature, at the same time if firing time equals to 1 hour and T=1150 ^{o}C then the specific resistance reaches the minimal value.

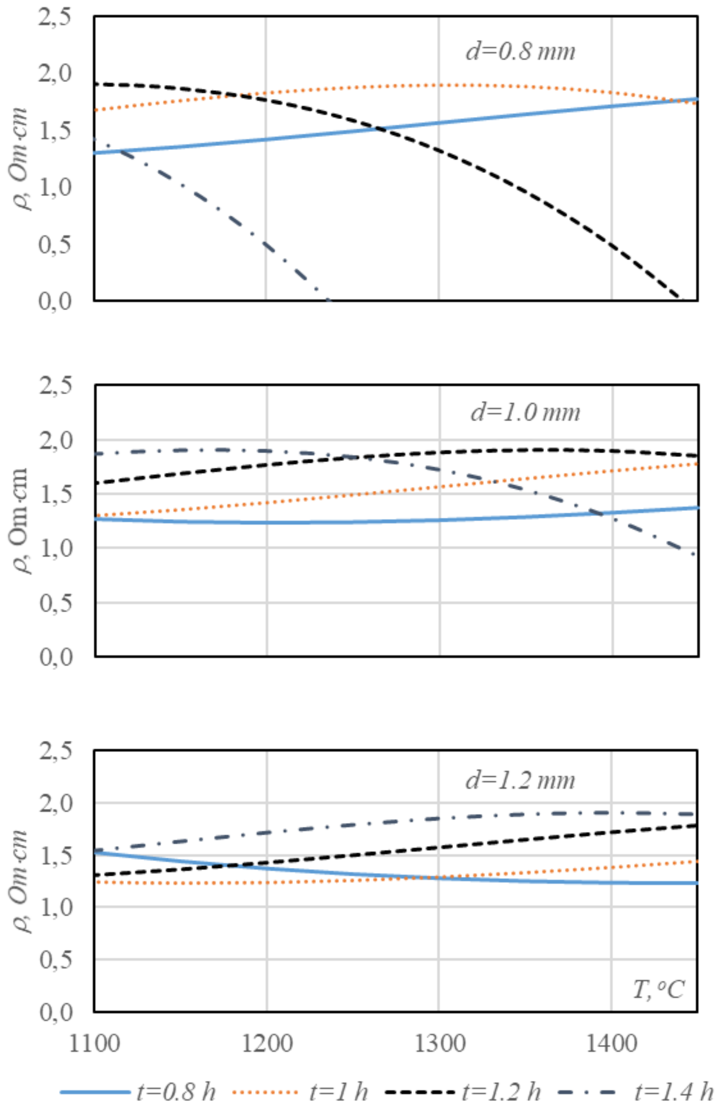
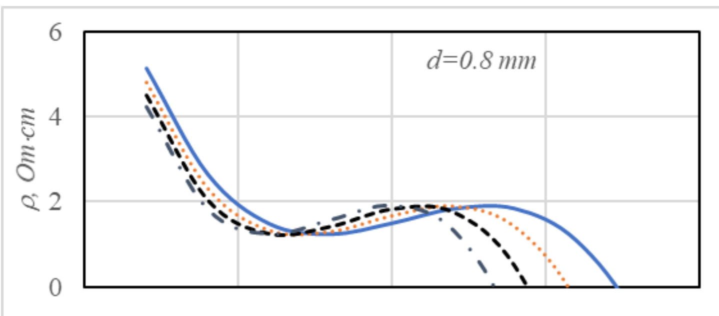


Figure 4. Dependence of the specific resistance on the firing temperature by the different widths and firing duration.

Dependence of specific resistance on the firing duration on case of plates width d (0.8 mm, 1 mm, 1.2 mm) is constant and in the different values of firing temperature T is presented on Figure 5. As we can see from Figure 5a that the specific resistance reaches the minimal value in d=0.8 mm, a) t=0.8 hours (T=1000 °C and 1100 °C), b) t=0.6 hour (T=1200 °C and 1300 °C). As to the Figure 5b, here in d=1 mm, a) t=0.8 hours (T=1000 °C), b) t=1 hour (T=1100 °C and 1300 °C) the specific resistance accepts the minimal value. When d=1.2 mm then the such minimal value of the specific resistance reaches in cases of a) t=1.2 hours (T= T=1000 °C) and b) t=1 hour (T= T=1300 °C) (see Figure 5v).



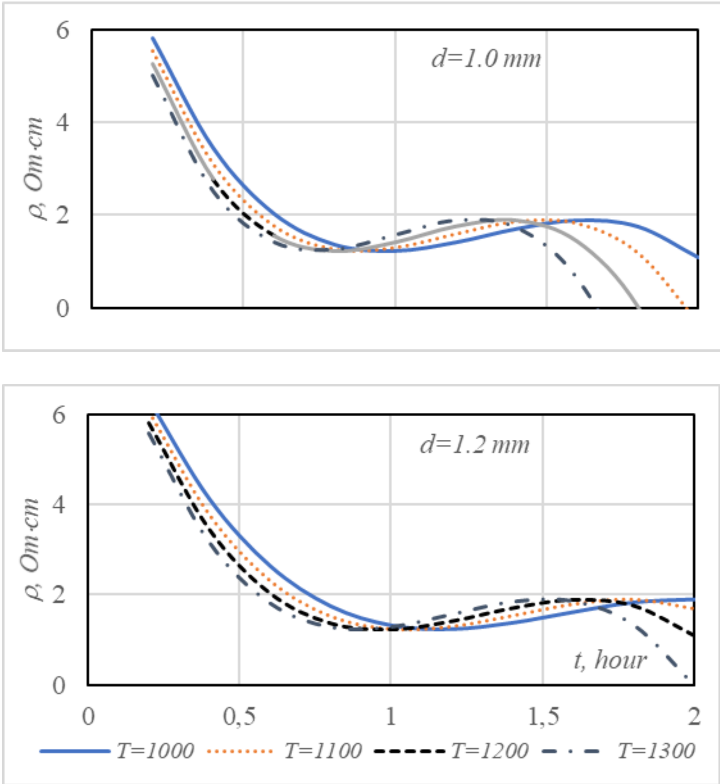


Figure 5. Dependence of the specific resistance on the firing duration by the different widths and firing temperatures.

4. Conclusion

Thus, in this paper we analyzed the experimental results on the features of PC silicon producing based powder technology and dependence of the specific resistance of obtained samples on the firing temperature, firing duration and samples width using "the smallest quadrates" method. The new unique representation for calculating the specific resistance of plates in dependence of firing temperature, firing temperature and samples width is obtained. This representation allows to choose two of these three parameters in case of arbitrary one is constant. That is when the firing duration is constant then ratio of the firing temperature to the plate width is constant too; if the firing temperature is constant then ratio of firing duration to the plate width is constant too; in case of constant of plate width produce of firing temperature to the firing duration is constant too. Moreover, the proposed ourselves representation allows to analyze theoretically the dependence of specific resistance on the third parameter in case of constancy of arbitrary two parameters.

Presented in this paper results can be used in future by young investigators working in this branch of science for producing PC silicon, based powder technology, and optimize its parameters.

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