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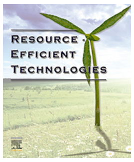

Research paper

# Obtaining the fine-grained silicon carbide, used in the synthesis of construction ceramics

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**Abstract**

Silicon carbide is used in the production of construction and temperature-resistant goods, capable of withstanding high mechanical and thermal loads. During recent times, silicon carbide has been frequently used in the electronics industry. Since sintered silicon carbide has increasingly been used as a replacement for metal components of various devices, the process of obtaining compact goods from silicon powder has become the defining factor in the technology used for its synthesis. The selection of conditions in which the sintering is conducted depends on granulometric structure, the form and the surface condition of the initial powder. The work consists of the synthesis of silicon carbide powder using the purified form of metallurgical silicon powder and soot. The qualities of testing samples were studied, where silicon carbide was obtained using established technology, from mechanically activated elementary, fine-grained silicon and soot, by pyrolytic synthesis. It was demonstrated that synthesis produces highly pure silicon carbide powder, (α- and β-phases) with a granulometric composition that allowed subsequent sintering to produce high quality compact goods. It was established that the content of silica in synthesized silicon carbide powder does not exceed 1–2% of the total mass.

*Keywords:* Silicon carbide; The Acheson method; Form and histogram of particle size

## 1. Introduction

Modern industrial machine building, metallurgical, chemical and energy sectors impose high standards for the construction materials used to make their components. Such requirements, which cannot be met by metals and alloys, are: high chemical resistance, ability to withstand high temperatures while keeping stable and, when exposed to extensive mechanical and thermal loads, low specific density, low production cost and reliance on environmentally friendly manufacturing technologies.

Leading industrial countries all recognise that ceramic is one such material, which meets all these requirements. The determination of the level of technological development in individual countries is based on the existence of industrial production potential for ceramic material and goods.

Silicon carbide is one of the most widely used materials. Classified as oxygen-free ceramic, its field of industrial application is constantly widening. This substance has unique qualities, such as high rigidity and stability, chemical and thermal stability, high melting point and resistance to oxidation, considerable resistance to erosion etc. Thanks to these qualities, SiC can be used to produce goods designated to withstand high electric power (power electronics) [1], to build electronic components that are resistant to high temperatures, as well as equipment exposed to high friction and heat [2]. Silicon carbide is used in the industry in the production of polyethylene (ethylene) for fittings and sleeves, allowing the process temperature to be doubled [3]. Silicon carbide is increasingly being used by nano-structures [4].

The main industrial method for the production of silicon carbide is SiC synthesis, which was conceived in 1892 by

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**Nomenclature**

| | |
|---|---|
| Q | the amount of heat released during reaction, $[\frac{kJ}{mole}]$ |
| D | particle size, in nm |
| σ | standard average deviation, in nm |
| $d_{95}^{cp}$ | average particle size, for particles constituting at least 95% of total particles, in μm |

[Edward Goodrich] Acheson. The process is based on a widely used method of $SiO_2$ recovery using the carbothermic process resulting in SiC powder. The main shortcoming of this process is the multitude of stages in technology, the high energy consumption and the rather low quality of the product (low purity). Several alternative SiC production methods have been reported [5,6]. They include physical splitting of volatile silicon compounds (PVT) [4,7], chemical decomposition of compounds (CVD) [8–12], the sol-gel process [13–15], liquid phase sintering (LPS) [16] and obtaining alloys through mechanical agitation (MA) [17].

The purpose of the present paper is to describe the principles of production of high purity SiC powder, to be used in the manufacturing of compact goods, capable of withstanding high pressure, temperatures and mechanical loads, as well as in [chemically] aggressive environments. For this we will need to study the effects of the granulometric composition of the initial silicon powder on aspects of the synthesised SiC powder particles. Based on the information provided under [18], the synthesis temperature has a decisive effect on the quality of SiC, which is why its correct setting is particularly important. When producing compact goods, one of the primary requirements is to ensure uniform sintering of aggregate SiC particles. This factor will depend on the condition of the sintered particle surface. The study of the effect of synthesis temperature and air annealing of the samples on their surface is the main purpose of the exercise.

It has been noted, that production of fine ceramics experienced an annual growth of 9.1% [19] and that by as early as 2000 this sector was already worth USD 8.45 billion. By 2015, the world's capacity for production of silicon carbide of all types kept increasing, churning out 1.1 million tons of goods per year, 41% of which was in China [20]. In addition to China, the growth in silicon carbide production was also recorded in India and Japan. The main consumers of silicon carbide are countries with developed metallurgical and electronic industries, where refractory ceramics are needed. In recent times, silicon carbide has also been used as a construction material, as well as being increasingly used in aviation.

In order to integrate silicon carbide further into industrial production, its quality and its physical qualities will need to be improved. With this in mind, production of construction materials from high purity silicon carbide attracts particular interest.

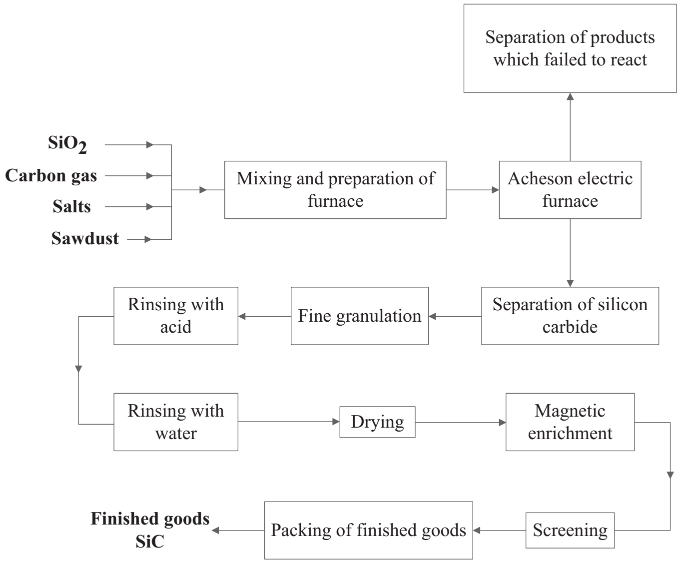

Fig. 1. A process flow diagram for the production of silicon carbide.

On an industrial scale, silicon carbide is produced [6,19] using the carbothermic method for the recovery of silica (the Acheson method) based on the following reaction:

$$SiO_2 + C \xrightarrow{>2000^\circ C} SiC + CO + CO_2. \quad (1)$$

Fig. 1 illustrates the principle of production of general purpose silicon carbide.

## 2. Experimental research and consideration of results

In the practice of manufacturing high construction ceramics it is recommended to use pure homogeneous fine-grained powders (sub-micron) of equidimensional composition, with a homogeneous form and equal reaction to sintering. This set of excellent qualities of silicon carbide powder (rather than individually achieved characteristics) guarantees high quality exceeding of similar goods produced by leading manufacturers.

Fig. 2 is a process flow diagram illustrating the synthesis of high-quality, miniature silicon carbide powders for use in construction [21]. The raw material, which is metallurgical powder, is cleaned of iron, aluminium and calcium impurities, based on methods described in scientific literature, to a purity of 99.95–99.97% mass for silicon [22]. The material is then treated with "drowned jet", on a machine equipped with a reactor, where it is finely powdered, mechanically activated and finally classified once it reaches the purified powder stage. Synthesis of the SiC-powder from previously mechanically activated powder Si is carried out at the maximum temperature of 1600 °C. In the industry the method of SiC-synthesis from the Si-powder without preliminary mechanical activation is applied; however, such process proceeds only at 1800–1900 °C, i.e. at a temperature at 300–400 °C above, than the offered process.

Only finely graded silicon powders from two groups are used in the synthesis, each with an average particle diameter of up to $d_{av} \le 1.0$ μm and $d_{av} \le 5.0$ μm, respectively, in a mass of up to

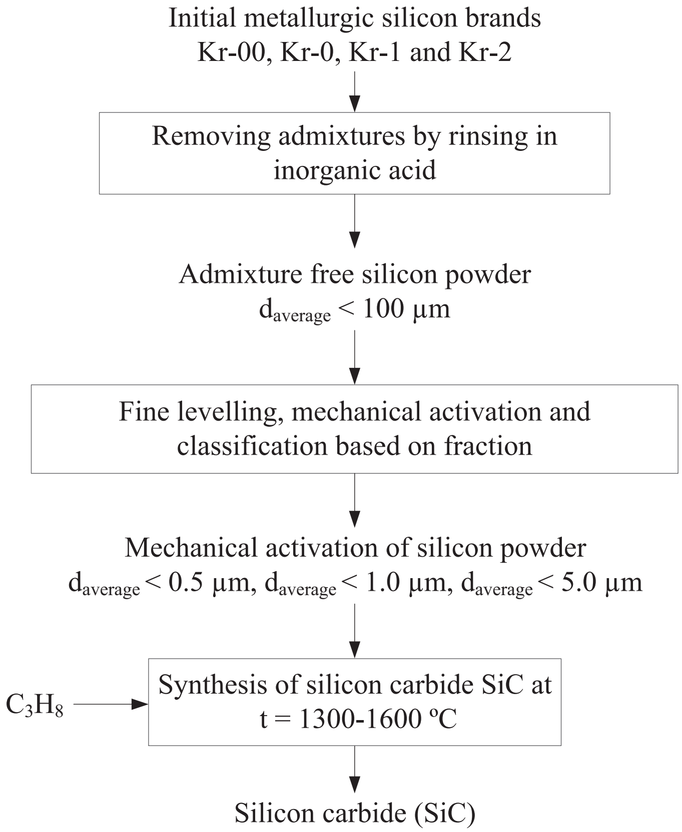

Fig. 2. A process flow diagram illustrating the production of fine grade silicon carbide powder.

200 g. The synthesis of silicon carbide is conducted in an electric vacuum furnace, from basic components – silicon and pyrolytically obtained carbon – in the ratio of 1:1 at 1300–1600 °C based on the following reaction:

$$Si_{(TB)} + C_{(TB)} \xrightarrow{1300-1600^{\circ}C} SiC_{(TB)} + Q. \qquad (2)$$

In the process of producing sintered goods, the form, in which components are submitted to synthesis, is very important. Where particles with a large diameter are sintered, for example, the dimensions can be significantly and uncontrollably reduced, to account for empty space around particles, and the disturbed form of the finished product, which are liable to further affect its characteristics. Consequently, morphology, granulometric composition, specific surface and phase constitution of silicon powder were studied in order to assess the possibilities of using powders of different particle forms and sizes, based on the diffraction electron microscopy, XRD-analysis and BET-analysis. These methods allowed us to predict the qualities of the final product obtained by sintering with a great degree of accuracy and in great detail [4].

Fig. 3 indicates that particles of silicon powder are mainly fine graded and flat and that their form is splintery. One of the reasons for the occurrence of dislocation substructures might be mechanical processing of silicon powder. Flat particles do not have intraphase and interphase borders, in other words, they are single phase monocrystals (Fig. 3a and 3b). In some cases

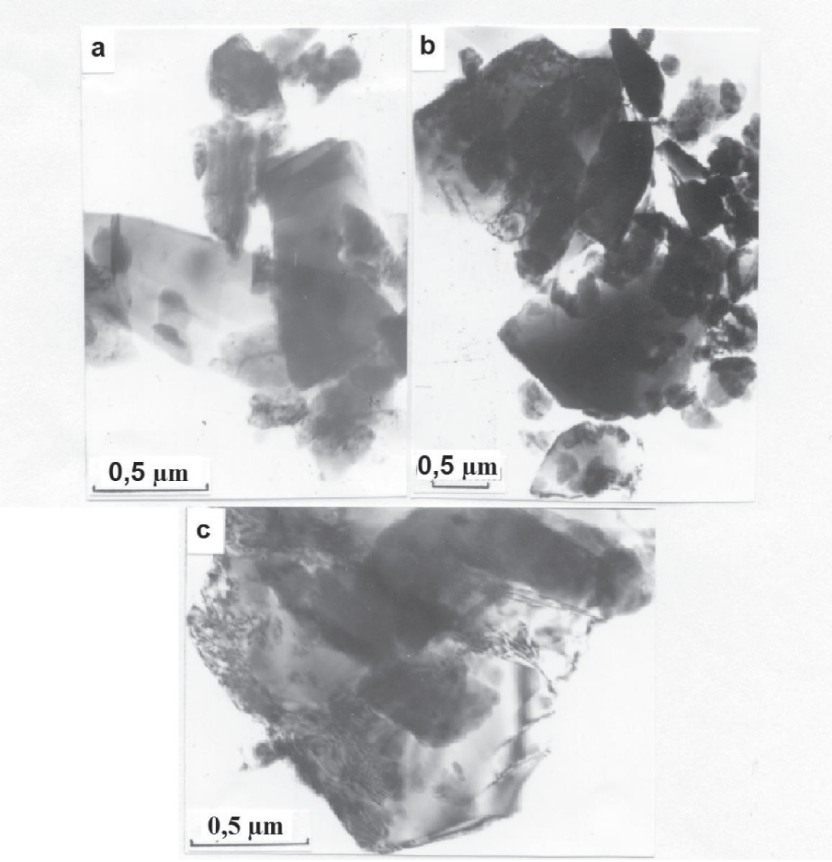

Fig. 3. The electron-microscopic image of silicon powder crystallites.

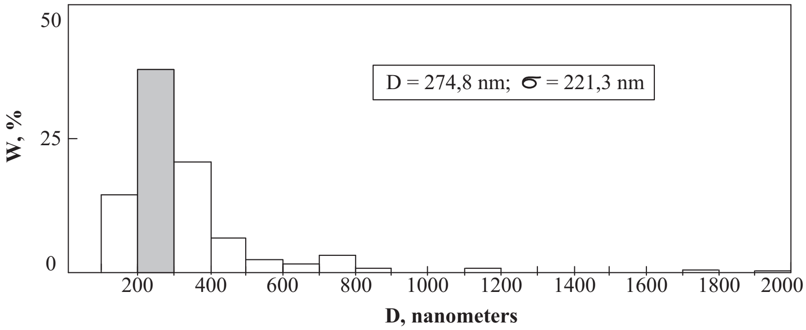

Fig. 4. Distribution of powder particles based on their size.

(Fig. 3c), flat particles had a defected substructure in the form of dislocation grids, mainly situated along the borderlines surrounding crystallites. This has a negative effect on the quality of the finished product in the process of sintering. It ought to be pointed out, that the presence of dislocation structure contributes to an increase in the ratio of diffusion of secondary components, accelerating the process of carburisation of material.

The histogram of particle sizes is given in Fig. 4, indicating their average dimensions (D) and their standard deviation (σ) The average particle size d = 274.8 nm (σ = 221.3 nm), the distribution of particles of silicon powder corresponds to normal dispersing. It ought to be pointed out that a certain volume of particles in the species were found to have dimensions that considerably exceeded (7 times) those of the main volume, however the quantity of these particles in the overall volume remained limited to 2–3%. Consequently, the effect of these particles on the forming of the sintered product can be ignored.

Fig. 5 indicates the results of XRD-analysis carried out on silicon powder specimens, obtained using the X-ray diffractometer Shimadzu XRD 7000 with scanning angles from 10 to 100°. We see a set of lines, characteristic for silicon, and silicon carbide. The oxygen content in silicon does not exceed initial values.

Definition of a specific surface of powder silicon particles is carried out by a BET-analysis method using the META SORBI-M analyser. For determination of specific capacity of a Si-powder monolayer, which is used for calculation of size of its specific surface the amount of the adsorbed nitrogen for values of relative partsialny pressure of h = $P/P_0$ equal 0.06, 0.09, 0.15, 0.2 is measured and dependence of values $f = \dfrac{P/P_0}{V(1-P/P_0)}$ on the relative partsialny pressure of h (Fig. 6) is constructed.

After the definition of an inclination tangent angle of a straight line $tg\alpha = \dfrac{f}{h} = \dfrac{0,2-0,09}{0,2-0,06} = 0,786$ and value *f*, which is defined when crossing a straight line with ordinate axis $V_m$ value is calculated:

$$V_m = \frac{1}{0,05+0,786} = 1,196$$

The size of a sample specific surface *S* is determined by the equation:

$$S = S_0 V_m = 4,34 \cdot 1,196 = 5,191\ \mathrm{m}^2/\mathrm{g},$$

where $S_0$ – the surface area which occupies 1 normal $\mathrm{sm}^3$ of the gas, adsorbed by a powder monolayer (for nitrogen $S_0 = 4,34\ \mathrm{m}^2/\mathrm{normal\ sm}^3$).

The synthesis of silicon carbide is conducted in a furnace, in argon atmosphere, at the prescribed temperature, for 4 hours.

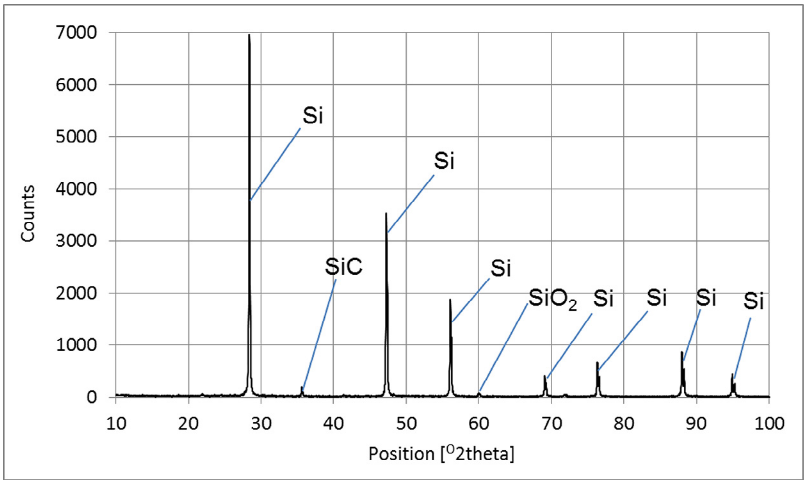

Fig. 5. The X-ray of a silicon sample, obtained based on the XRD-analysis method.

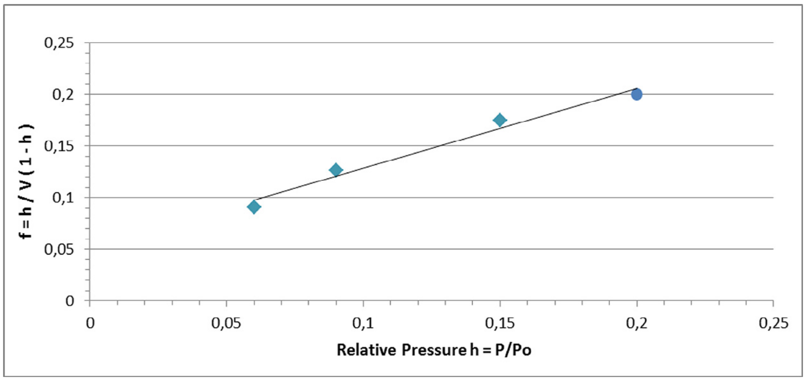

Fig. 6. The dependence of variation in the degree of saturation *f* on relative pressure h.

Once the furnace is cooled, the synthesised silicon carbide powder undergoes thermal processing in order to remove excess carbon, in an open electric furnace, for 8 hours, moving the specimens once every 4 hours. The analysis was conducted on granulometric composition and phase constitution of silicon carbide samples. Earlier research indicated that the synthesis of silicon carbide is best conducted at 1300 °C. Consequently, the tested silicon carbide specimens were synthesised in the prescribed conditions:

1. 1300 °C in argon atmosphere without aerial annealing
2. 1600 °C in argon atmosphere without aerial annealing
3. 1600 °C in argon atmosphere with aerial annealing

## 3. Silicon carbide specimen, synthesised at 1300 °C

The results of electron-microscopic analysis, indicated in Fig. 7, established that synthesised SiC powder has the following phase composition: α-SiC, β-SiC, $SiO_2$ and graphite. It ought to be pointed out, obtaining the images in different modes, allows us to identify different size particles. Consequently, for α-SiC, large fractions become visible on a clear background (using the underlying light), while small particles are visible on a dark background. Morphology of particles – sphere. Particles α-SiC (Fig. 7c, d) and β-SiC (Fig. 7a) are laid out as conglomerates, sized 0.1–0.2 µm and over.

The analysis of the granulometric composition of β-SiC indicated (Fig. 8a), that the main mass of particles falls within a narrow dimensional scope, measuring a mere 6,25 nm, standard deviation σ = 2,23 nm, so these conditions ensures continuity of the characteristic of the material in terms of volume. The α-form of SiC has much larger particles, the average diameter of which is 20,5 nm, standard deviation σ = 12,7 nm (Fig. 8b); however, their size range tends to be wider than that of β-SiC. Heterogeneity of the particles sizes α-SiC about 12,7:2,35 = 5,4 times more, than β-SiC. Therefore for receiving products which properties have to be identical on all volume it is necessary to use the β-SiC. Graphite particles (Fig. 8c) have hexagonal and cubic crystalline grids; these particles are either dispersed in small clusters or remain separated. Their size can change from 20 to 160 nm, the average diameter of their main

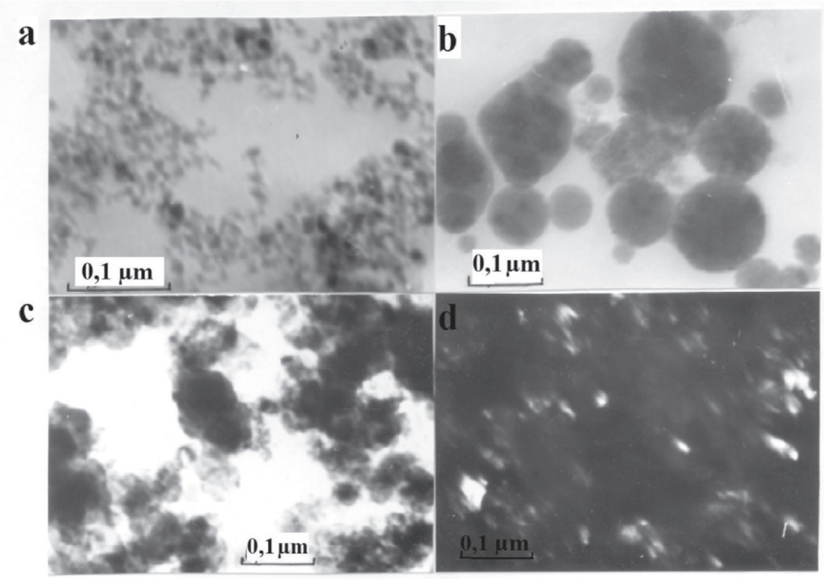

Fig. 7. The electron-microscopic images of silicon carbide crystallites (SiC): a – β-SiC; b – C-graphite; c – α-SiC (in the clear area); d – α-SiC (in the dark area).

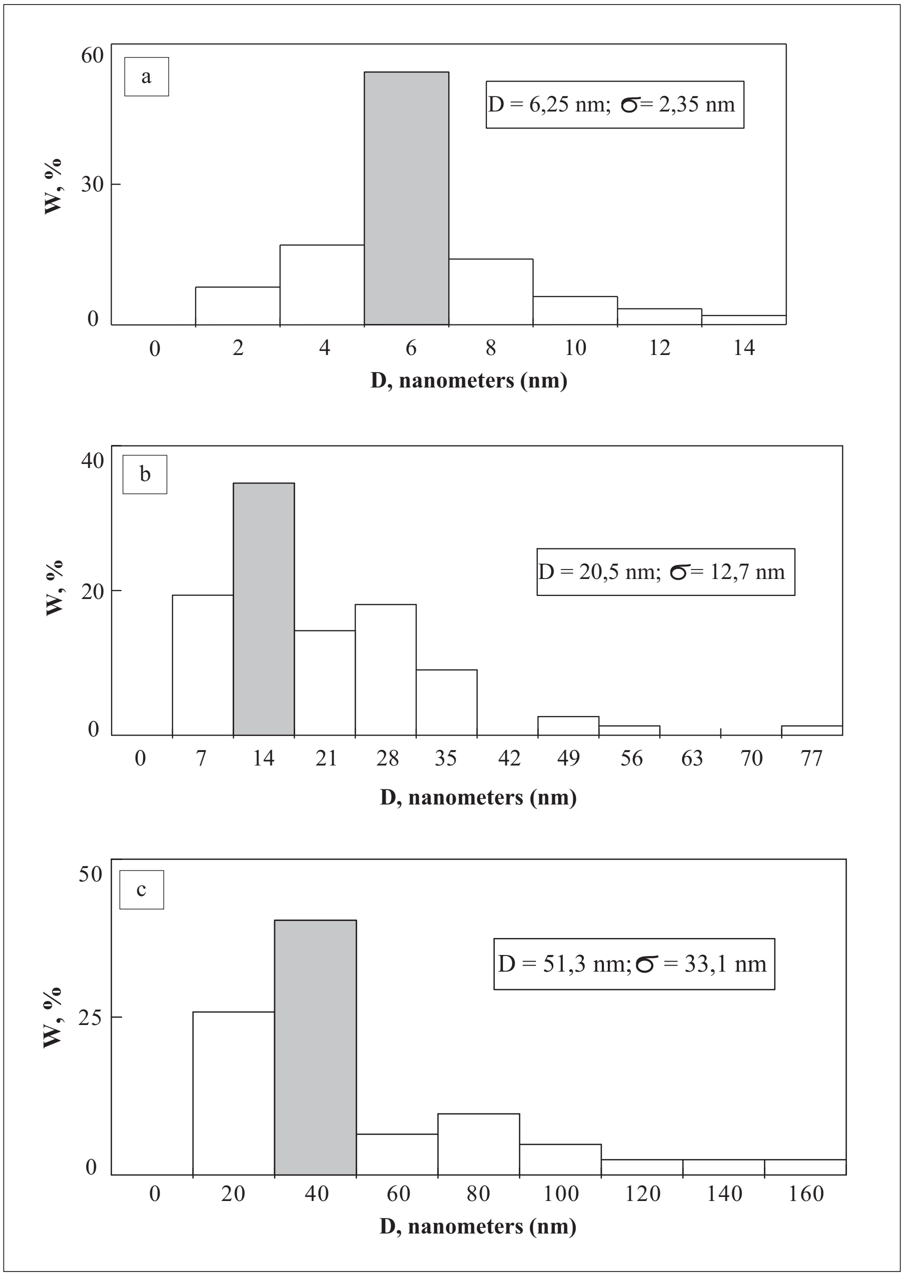

Fig. 8. a. Histogram of sizes of β-SiC particles; b. Histogram of sizes of α-SiC particles; c. Histogram of sizes of graphite particles.

mass (~40%) being 51,3 nm, standard deviation σ = 33,1 нм. Therefore in the course of SiC-synthesis small particles C (51,3 nm) will be occluded on a surface of large particles of Si (274,8 nm,); as a result, the size of the formed particles of SiC changes slightly.

Specimen for carrying out chemical synthesis contains insignificant quantity of $SiO_2$ (Fig. 5) therefore in all the synthesized SiC-specimen silica ($SiO_2$) is found in the form of spherical particles along with carbide phase particles. It has been impossible to separate silica particles, since the part of it that has not reacted to synthesis, is subject to considerable pollution by carbon, which is present in the initial mixture; this is explained by the good absorption capacity of $SiO_2$. Based on the frequency of occurrence of individual phases, the following assessment of their content was produced as part of the indexing, based on diffraction electron microscopy of samples: silicon carbide ~70% (α-SiC – ~47%, β-SiC – ~23%), graphite ~23%, and silica ~7%. It ought to be pointed out, that the partition of the carbide phase into α- and β-modifications, using electron microscopy, is not entirely justified, since the frequency of corresponding (most intensive) analytical signals for each given modification are close, in terms of intensity, which affects the sensitivity scope of microscopy imaging.

## 4. Silicon carbide specimens, synthesised at 1600 °C, without annealing and those with aerial annealing

Using the diffraction electron microscopy XRD-analysis, we examined the form, size and composition phase of silicon

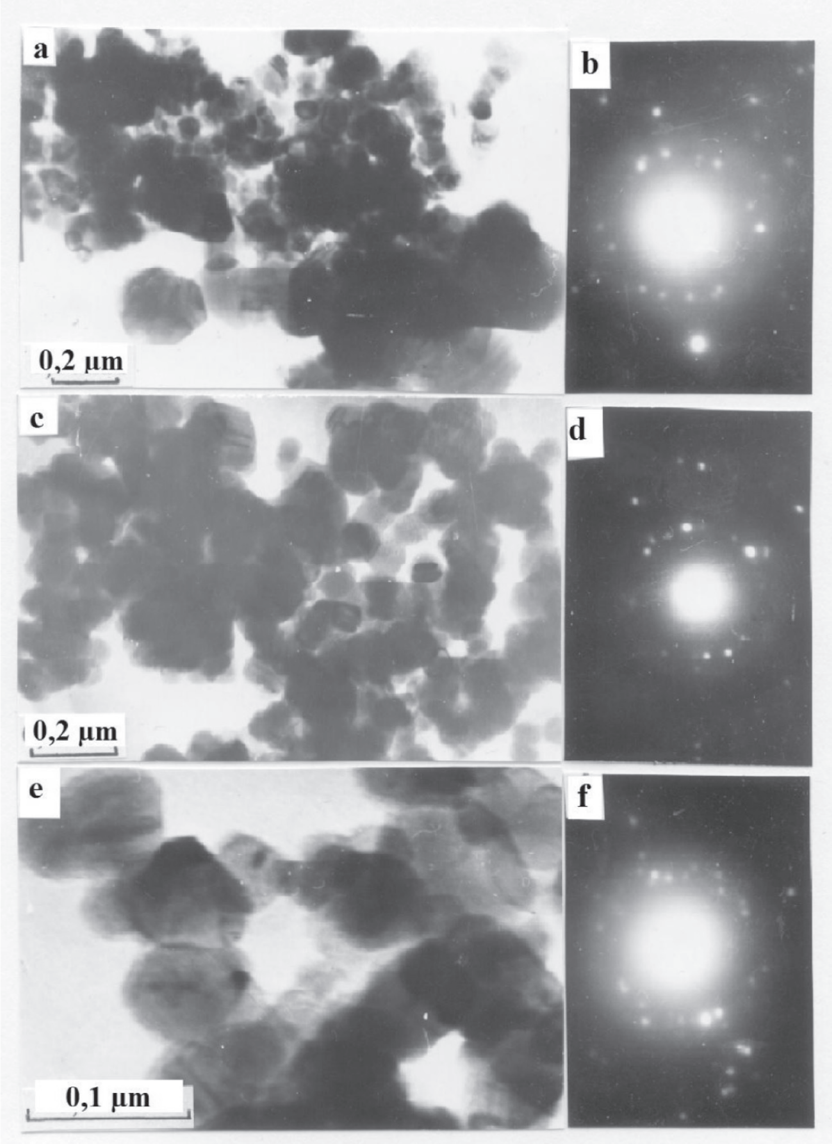

Fig. 9. Electron-microscopic images of SiC powder particles not treated with aerial annealing: a,c,e – light blue images of SiC particles, with the size falling within the nanometric range; b,d,f – diffraction electron microscopy images of the given particles.

carbide powder, without annealing and with aerial annealing. Three particle specimens were synthesised, and their enlarged microscopic images indicated in Fig. 9. The heating intensity of the initial mix of silicon and graphite was 0.3, 0.6 and 0.9 °C/minute, during the synthesis.

The analysis of granulometric composition of the samples indicated that the diameter of all synthesised particles was within the nanometric scope. In Fig. 10a (1st sample), we note that at the heating intensity of the initial silicon and graphite powders, equal to 0.3 °C/minute, the samples obtained fall within a narrow size range – from 50 to 250 nm – with most particles being 145,4 nm, (standard deviation $\sigma = 74{,}3$ nm); this indicates that the conditions during the synthesis were stable.

The main quantity of the 2nd sample particles (heating intensity of initial mix of the Si and C powders equal to 0.6 °C/minute) is having the size of 45,7 nm and standard deviation $\sigma = 19{,}2$ nm. The synthesized particles of the 2nd SiC-sample are 145,4:45,7 = 3,2 times smaller than those from the first specimen (Fig. 10b). Thus, if the heating intensity of initial powders increases, then the size of SiC-particles considerably decreases.

The 3rd SiC-sample was synthesized at a heating intensity of the Si and C powders mixture, is 0.9 °C/minute; the size of the formed SiC-particles changes in the wide range from 10 to 450 nm; the main quantity of particles has the size of 94.6 nm, a standard deviation $\sigma = 72.8$ nm. The size of the SiC-particles of the 3rd sample is 145.4:94.6 = 1.5 times less than the first, but is 94.6:45.7 = 2.1 times more than the second (Fig. 10c). These results show that at a very high speed of heating of initial powders because of unstable conditions in which the synthesis was carried out, as well as quick mixing of initial components at the outset of the process, which prevented the creation of uniform crystal structures within the reaction range.

Each of 3 samples synthesized at 1600 °C has aerial annealing (Fig. 11). At the 1st annealed sample. In the group of particles that was treated with annealing, inspection of the

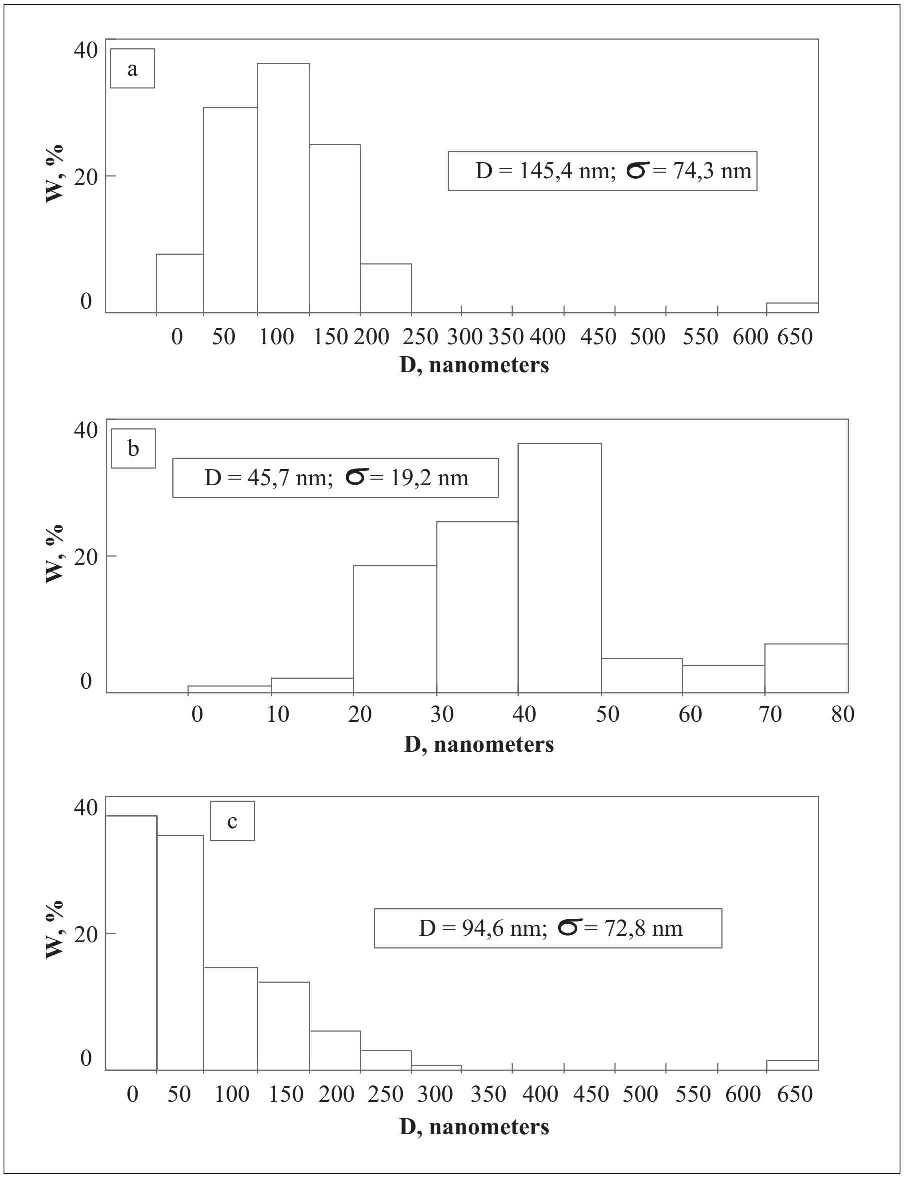

Fig. 10. Dispersion of silicon carbide particles not treated with aerial annealing. Heating intensity of the initial powders Si and C: 1 – 0.3 °C/minute; 2 – 0.6 °C/minute; 3 – 0.9 °C/minute.

particle interior revealed a striated contrast, similar to that found in micro-twins (Fig. 11a). Particles of both samples mainly have polyhedron form and are less frequently rounded. At the 2nd and 3rd samples, the particles could be divided into two groups, nano-particles and submicroparticles, both having similar volume ratios. (Fig. 11c, 11e). Microdiffraction patterns of samples show that the particle sizes of the 3rd sample (Fig. 11f) is more than the 2nd (Fig. 11d), but is less, than first (Fig. 11b).

Fig. 12 Histograms of particle sizes. In all samples, the quantity of large particles, in comparison with samples has increased to annealing (Fig. 10).

After annealing, the size of the particles of the 1st sample changes from 100 to 450 nm, and the main quantity of the particles is 190,8 nm, with a standard deviation $\sigma = 75$ nm. In the course of annealing the quantity of the particles having the size of 250 nanometers has increased by 20:8 = 2,5 times; 5 and 7% of particles which size is equal to 300 and 400 nanometers, respectively (Fig. 12a) were formed. In the 2nd sample, after annealing, the main quantity of particles have the size of 55,1 nm, with a standard deviation $\sigma = 22{,}1$ nm; ~9% of the particles have the size of 97 nm (Fig. 12b). The main quantity of particles in the 3rd sample, after annealing on air had the size of 122,3 nm; standard deviation $\sigma = 87{,}4$ nm; ~5% of the particles have the size of 370 nm (Fig. 12c). The analysis of results indicated that annealing of SiC powder, leads to a slight enlarging of the particle in both size groups.

The SiC-sample synthesized by heating of silicon and graphite powders with a heating intensity of 0.3 °C/minute has the most stable characteristics.

The XRD analysis of this sample by means of the Shimadzu XRD 7000 diffractometer at the angles of scanning is executed from 10 to 100°.

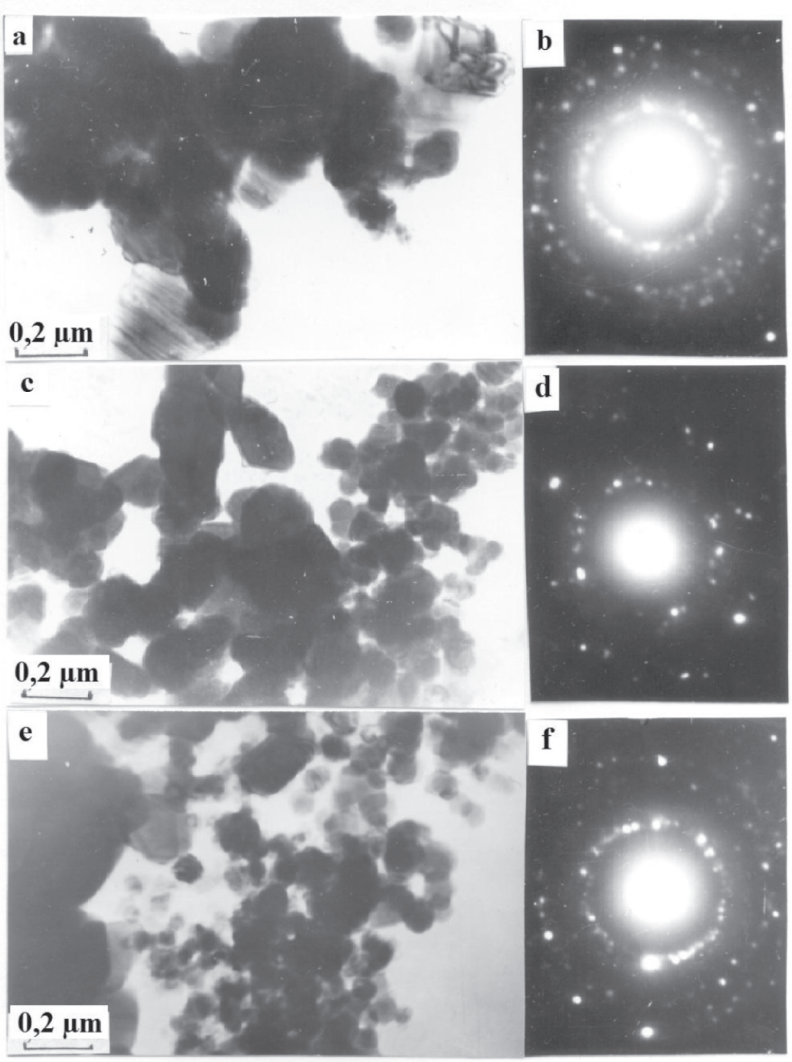

Fig. 11. The electron-microscopic images of the part of SiC powder individual treated with aerial annealing: a – particles whose dimensions are under 1 micron; b,c – particles of two size ranges; a,c,e – light blue images; b,d,f – electron-microscopic images.

Fig. 13 indicates the results of XRD-analysis carried out on silicon carbide powder specimen, obtained using the X-ray diffractometer Shimadzu XRD 7000 with scanning angles from 10 to 100°. We see the two lines, characteristic for silicon, and silicon carbide.

The lines characteristic of SiC-powder are well identified and not imposed on the line characteristic of Si-powder. SiC is identified in specimen by measurement of line intensity for the angles of scanning 34°, 36°, 38°, 42°, 60°, 72°. The $SiO_2$ content in SiC does not exceed initial values.

## 5. Conclusions

The technology described in the present paper on silicon carbide samples was obtained from different synthesis conditions. The subject of the analysis was the particle's phase and granulometric composition. With this in mind, using micro diffraction analysis and consequent indexing of electron images, it was indicated that the main phase of the tested samples was silicon carbide (SiC), which was found in both α- and β-modifications. Silica constituted an additional phase ($SiO_2$). No free silicon or graphite particles were detected. In samples not treated with annealing, the submicron particles were mainly those from the β-SiC category. Nanometric particles represented a mix of α- and β-SiC. Silica was found in clusters of these particles. The reflexes of the silica crystalline grid were relatively weak, indicating a low (~1–2%) volume ratio for this phase.

In the samples treated with aerial annealing, α- and β-modification of SiC were noted in both size groups. Silica was found in both nanometric and submicron size groups. The volume ratio of silica for particles not treated with annealing was ~5%. Based on requirements set for powders used in construction, the given content of $SiO_2$ is acceptable and does not affect the quality of sintered goods.

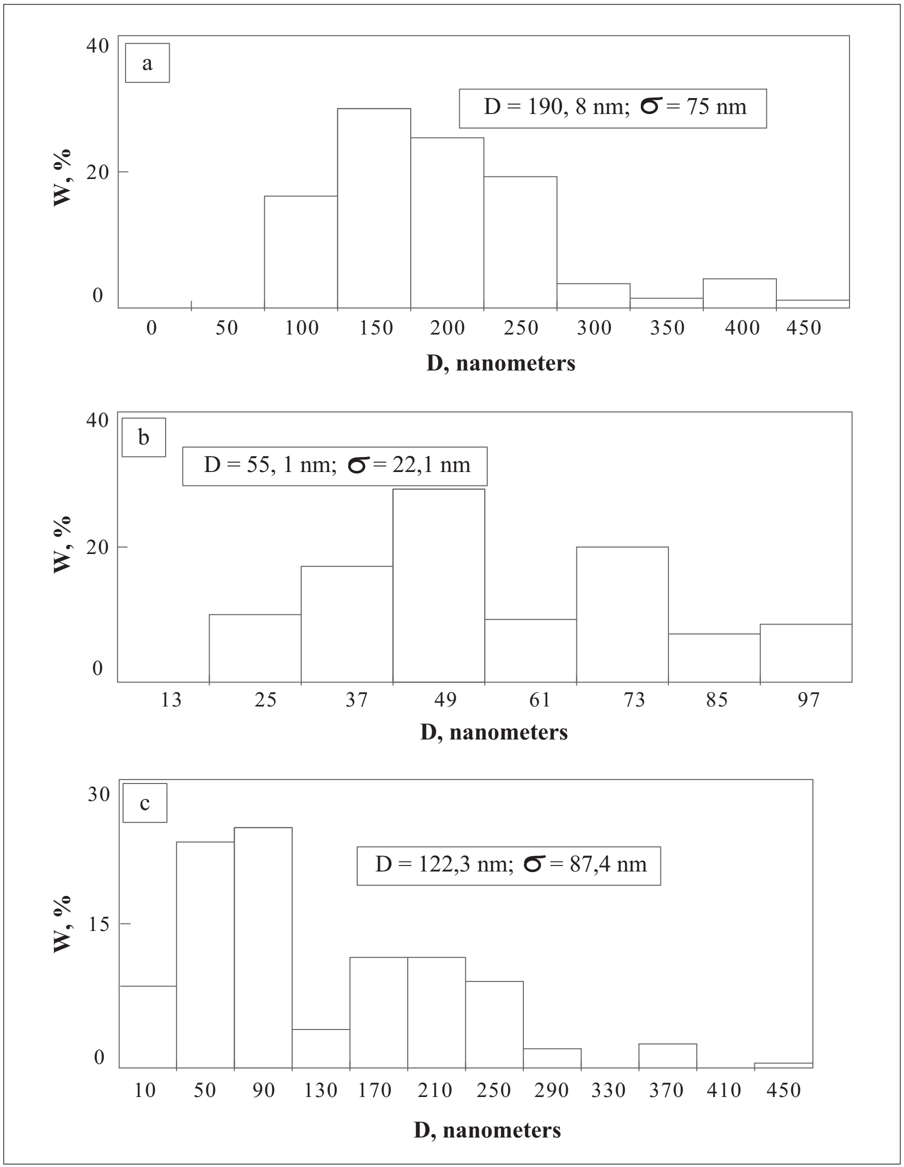

Fig. 12. Dispersion of silicon carbide particles, based on size, after completion of aerial annealing: a – histogram of particles whose size is under one micron; b – histogram of particles whose size falls within the nanometric range; c – histogram, of particles not divided into size groups.

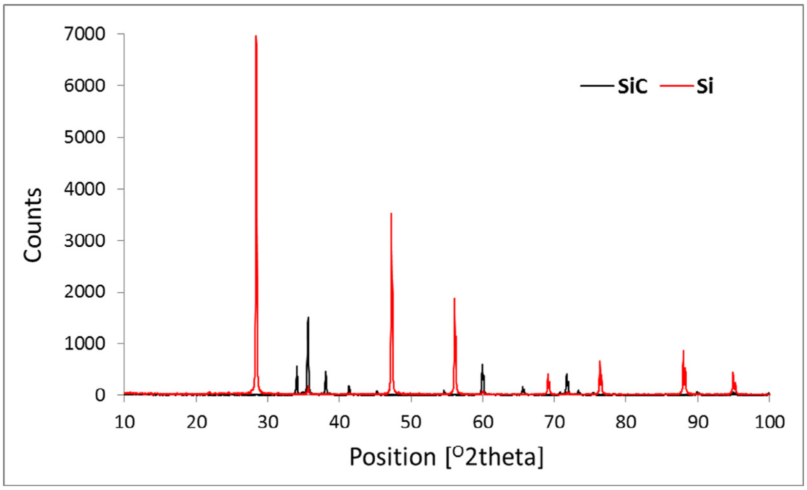

Fig. 13. The X-ray of a silicon carbide and silicon specimens, obtained based on the XRD-analysis method.

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