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The Chemical and Mineralogical Studies of Microsilica for Obtaining Silicate Materials

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ARTICLE INFO	ABSTRACT
Published Online: 24 August 2023 Corresponding Author: **Niyazova Sh. M.**	In this scientific work, the chemical and mineralogical properties of microsilica formed during the production of ferroalloy in the production of silicate materials at JSC “Uzmetkombinat” were studied. It has been established that the obtained results of the analysis confirm the technological feasibility of using microsilica in silicate materials, in particular for the production of glass, heat–insulating, oxide ceramic materials, as well as ceramic pigments.

KEYWORDS: silicate materials, microsilica, ferroalloy, pulverized waste, phase transformations, X–ray phase analysis, electron microscopic analysis, crystal structure, X–ray amorphous.

INTRODUCTION

The development of the production of silicate materials in the Republic of Uzbekistan is inextricably linked with the intensive development of the construction complex and other industries [1]. The development of new highly efficient and energy–saving compositions also remains an urgent task for researchers working in this field [2, 3]. In this regard, the initial interest in the use of production waste in silicate materials was due to environmental problems, increased control of atmospheric pollution, as well as the need to save energy in the building materials industry by partially replacing natural materials with industrial waste [4].

It is known [5, 6] that large resources of dusty wastes from the production of silicon and high–silicon ferroalloys containing a significant amount of silicon dioxide predetermine the relevance of the problem of their rational use. However, at present, most enterprises are further accumulating microsilica. This leads to economic losses associated, on the one hand, with the non–use of industrially valuable waste, on the other hand, with the cost of their storage. In addition, as a result of disorderly collection and accumulation, dust waste often loses its value as a raw material for possible processing, and currently existing methods for storing microsilica are not environmentally friendly. Therefore, the recycling and use of dust emissions should be considered as an important direction of saving material resources, as well as improving the efficiency of environmental protection in the production of silicon ferroalloys.

As a result of the analysis of published works, it was revealed that silicon oxide (silica) is contained in a large amount of large–tonnage industrial waste, in particular, mining waste contains 20–80% SiO_2 [7, 8], metallurgical waste 16–55 % [9], in ashes and ash and slag of thermal units is 27–63% [10]. Its highest content is found in waste products from the production of ferroalloys, which is up to 92%.

Dust wastes from the production of ferroalloys are condensed aerosols and, according to the classification, are classified as fumes. To date, the following terminology has been observed that combines this type of waste “Silica powder” (silica powder), microsilica, silica fume (silica dust entrainment, silicate smoke). It should be noted that the term “microsilica” is usually used in domestic practice [11].

Microsilica is a condensed silica dust that is a by–product of the production of crystalline silicon or silicon alloys by reducing quartz in an electric furnace. During the smelting of silicon alloys, some of the silicon monoxide SiO_2 passes into a gaseous state and, subjected to oxidation and condensation, forms an extremely fine product in the form of spherical particles with a high content of amorphous silica [10].

Thus, a significant number of works of domestic and foreign authors are devoted to the study of the properties of microsilica produced by ferrosilicon. The physico–technological characteristics of microsilica have been most fully studied in order to study the possibility of its use in the construction industry, the most traditional area [12]. Physical and chemical assessment of microsilica produced by ferroalloys is also usually applied in nature and is carried out mainly by chemical composition and fineness.

In the production of ferroalloys, silicon oxide, in the form of microsilica, is accompanied by the formation of large amounts of dust waste, which are carried out with gaseous products. At the same time, in the example of Uzmetkombinat JSC, the specific amount of emissions in the form of microsilica dust formed during the production of ferrosilicon grades FS45 is 0.465 g/t. The silica content in dust waste usually increases with an increase in the silicon content in the alloy [13] and is ~ 60% when smelting low–percentage grades of ferrosilicon (FS18–FS45) and up to 90% when smelting high–percentage grades (FS65–FS75).

The process of formation of microsilica occurs due to the mechanical entrainment of fine fractions of the charge for the production of ferrosilicon. The formation of microsilica as a result of oxidation reactions in gas ducts, occurring mainly in oxidizing furnaces, can be described as follows:

$2SiO$ (gas) + O_2 (gas) = $2SiO_2$ (solid)

Si (gas, solid) + O_2 (gas) = SiO_2 (solid)

SiC (solid) + $2O_2$ (gas) = CO_2 (gas) + SiO_2 (solid)

In this regard, the possibility of using microsilica, which is formed during the production of ferrosilicon at Uzmetkombinat JSC, for the synthesis of silicate materials has been studied.

MATERIALS AND METHODS

To determine the crystalline phase of the experimental samples of microsilica used X–ray phase analysis. X–ray phase analysis of sample samples was carried out by the powder method on an X–ray diffractometer brand LABX XRD–6100 SHIMADZU in the range 2θ, 10–80 using CuKα radiation with a wavelength of 1.5418 Å. X–ray photographs were taken with a step of 0.02 deg, voltage 30 kV, current 30 mA. In calculations and in the identification of crystalline phases, international standard reference data were used [14, 15].

The study of the structural characteristics, in particular, the shape and size of microsilica particles, was carried out using the methods of electron microscopic analysis. The images were obtained on a scanning electron microscope SEM MA 10, Carl Zeiss, using back–scattered electrodes Signal A = SE 1, under the shooting conditions: voltage ENT–15.0 kV, working distance WD–8,5 mm. The description of the crystalline phases and interpretation of the structural characteristics of the prototypes was carried out using a reference book [16, 17]. Preparation of preparations was carried out as follows. Microsilica powder was sprayed onto a pre–rolled indium plate 10x8x1 mm in size, the sawn layer was pressed in, and the residues were removed by blowing the surface. Next, particles impregnated into a metal matrix were studied. In the study of powders, the increase was up to 300,000.

RESULTS AND DISCUSSIONS

The chemical composition of a microsilica sample formed during the production of ferrosilicon at Uzmetkombinat JSC depends on the grades of alloys being smelted and was determined using standard methods of silicate technology [18, 19]. It has been established that the content of the main phase–silica in ferrosilicon waste is 85.0–89.0 wt.%. Free carbon in silica fume 0.5–1.2 wt.%, free silicon in the amount of 0.12–0.18 wt.%. Loss on calcination of microsilica at a temperature of 1000°C is 4.5–6.7 wt.%, which is consistent with the results of differential thermal analysis. In the chemical composition of microsilica samples, there are also free chemical elements in the form of carbon and silicon, the remaining elements are predominantly in the oxide form. The results of the study of the chemical composition of silica fume are shown in Table 1.

The formed microsilica is a fine gray powder. Microsilica samples of ferrosilicon gas cleaners are characterized by low bulk density, which is 0.25–0.32 g/cm^3. The fractional composition of the studied samples is in the range of 0.0006–0.0010 mm, depending on the alloy grade being smelted. The moisture content of microsilica, depending on storage and atmosphere, is in the range of 1.5–3.0%.

Table 1. The results of chemical analysis of samples microsilica JSC “Uzmetkombinat”

Sample name	The content of oxides per air–dry matter, wt, %						LOI, wt.%
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	
MS–1	85.0	0.7	1.1	3.5	2.7	0.4	4.5
MS–2	86.5	0.8	1.3	3.8	2.8	0.5	5.8
MS–3	89.0	0.9	1.5	4.1	3.0	0.6	6.7
Average sample	86.8	0.8	1.3	3.8	2.8	0.5	5.7

Loss on ignition (LOI) includes: water, organic and volatile impurities and carbon dioxide (CO_2).

It was established in [20] that the adsorption moisture capacity of a microsilica sample is relatively low; therefore, the amount of water required to create one molecular layer of microsilica is $6.336 \cdot 10^{-3}$ g/m^2. The number of water monolayers adsorbed on the particles is 3–4. At the same time, this amount of water is sufficient for the formation of a gel–like phase on the surface of microsilica particles, which increases adhesion. The hydraulic activity of microsilica, estimated by the amount of lime absorbed from a saturated solution at a temperature of 85°C, and the water demand are 100–104 kg CaO/t and 40–42%.

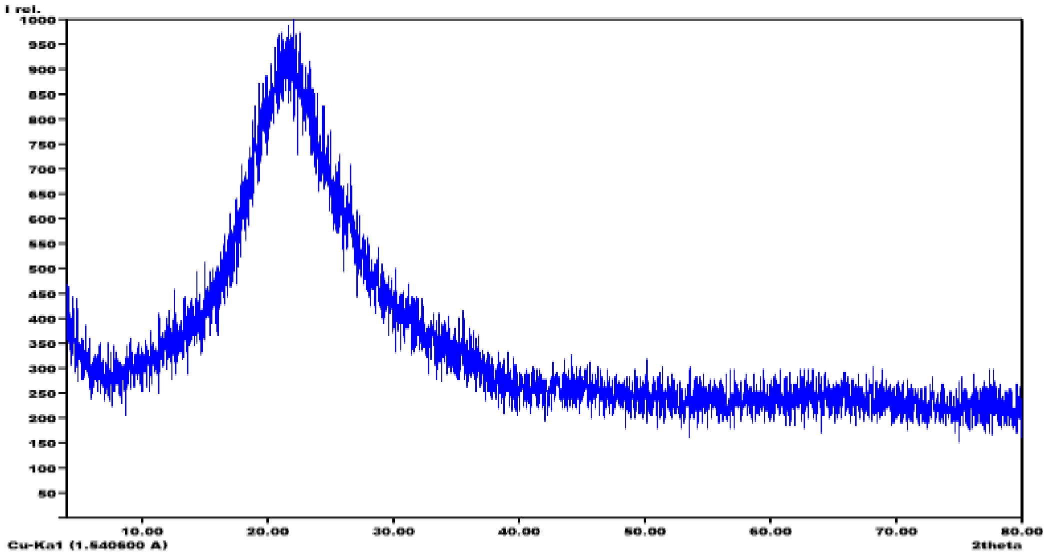

Figure 1. X–ray pattern of microsilica–a waste product of the production of ferroalloy JSC "Uzmetkombinat"

As a result of the identification of X–ray patterns (Figure 1) of the original microsilica, it was found that the phase composition of microsilica is X–ray amorphous. Therefore, the X–ray diffraction pattern of a microsilica sample causes difficulties in phase identification, which is also noted in [21].

In this regard, for the need to determine the crystalline components in the mass of silica fume, the magnetic fraction was demagnetized. Based on the results of identification of the phases of the X–ray diffraction pattern, it was established that the obtained magnetic fraction of silica fume consists of crystalline silicon, magnetite, and hematite. The X–ray pattern also revealed minor diffraction peaks related to the mineral α–quartz, α and β–silicon carbide (Table 2).

In the non–magnetic fraction, silicates of the type α–$CaSiO_3$, $Al_2O_3 \cdot SiO_2$, $Ca(Mg,Fe)_3(SiO_3)_4$ and silicon carbide were identified, while in the heavy fraction containing a small amount of the crystalline phase, only silicon carbide was determined.

Table 2. X–ray phase characteristics of the magnetic fraction of silica fume produced by ferrosilicon JSC "Uzmetkombinat"

Interplanar spacing, d/n, nm (2Θ, CuKα)		Intensity of diffraction maxima, I, %	Mineralogical composition of crystalline phases of magnetized microsilica				
d, Å	Q, nm						
3.31	0.3312	17	–	–	–	–	α–quartz
3.19	0.3191	96	–	Si	–	–	–
2.99	0.2992	42	Fe_3O_4		–	–	–
2.72	0.2718	21	–	–	–	Fe_2O_3	–
2.55	0.2554	100	Fe_3O_4		SiC	Fe_2O_3	–
2.11	0.2113	14	Fe_3O_4		–	–	–
1.96	0.1955	15	–	Si	–	–	–
1.68	0.1676	16	–		–	Fe_2O_3	–
1.63	0.1627	30	Fe_3O_4	Si	–	–	β–cristobalite
1.53	0.1532	14	–	–	SiC	–	–
1.49	0.1492	25	Fe_3O_4	–		–	–

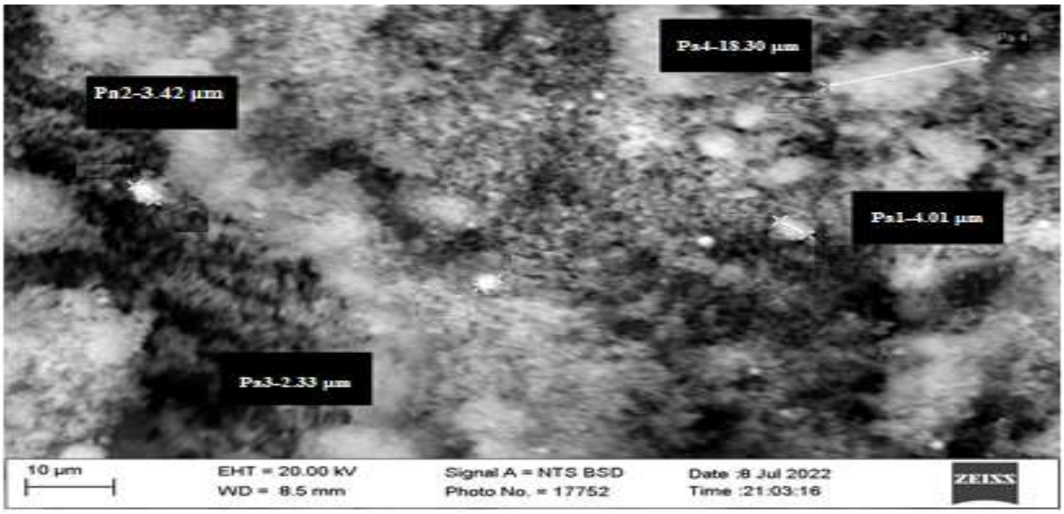

Figure 2. The electron microscopic images of microsilica

The results obtained by SEM methods make it possible to track the morphological structure of aggregates, the shape and size of individual microsilica particles. In general, it shows (Figure 2) that microsilica formed during the production of ferrosilicon consists of particles of a predominantly spherical shape with a size of 0.1–0.4 µm, combined into aggregates up to 1 µm in size. In some regions of the spectrum, the increase in particle size is due to the preparation of samples–preparations for electron microscopic studies.

CONCLUSION

Thus, the main characteristics of silica fume formed during the smelting of ferrosilicon produced by JSC “Uzmetkombinat” were studied to establish the possibility of their integrated use in a number of industries, in particular, first of all, in the construction industry, in the rubber industry, in the production of refractories and silicate materials, in metallurgy.

The results obtained confirm the technological feasibility of using microsilica in various industries of the Republic of Uzbekistan.

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