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Silica nanoparticles produced by DC arc plasma from a solid raw materials

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Abstract. Plasma synthesis of SiO_2 nanoparticles in experimental atmospheric pressure plasma reactor on the basis of DC arc plasma generator was presented in this paper. Solid high-silica raw materials such as diatomite from Kamyshlovskoye deposit in Russia, quartzite from Chupinskoye deposit in Russia and milled window glass were used. The obtained nanoparticles were characterized based on their morphology, chemical composition and size distribution. Scanning electron microscopy, laser diffractometry, nitrogen absorption (Brunauer–Emmett–Teller method), X-ray photoelectron spectroscopy and energy-dispersive X-ray spectroscopy were used to characterize the synthesized products. The obtained silica nanoparticles are agglomerated, have spherical shape and primary diameters between 10-300 nm. All samples of synthesized nanopowders were compared with commercial nanopowders.

1. Introduction

At present time due to the increasing need for nanosized oxides with special properties to modify different materials the problem of effective production technology developing is particularly relevant. There are a broad range of nanoparticles synthesis methods such as sol-gel, laser ablation, mechanochemical, plasma and flame processes [1-7]. Each of them has some prospects and constraints, but top-priority direction of investigations is the creation of low-waste and economical technology, such as plasma chemical synthesis. Silica nanopowder is one of the most widely applied additives in industry. As a rule, it helps to increase mechanical and chemical properties of materials [8-12]. But nowadays there are not so many works concerned with using low-cost solid raw materials for production nanosilica.

The paper mainly focuses on the investigation of SiO_2 nanopowder obtained from high-silica raw materials by using energy of cold plasma.

2. Materials and methods

Three types of high-silica were used as raw materials for plasma synthesis of silica nanopowder: quartzite (Chupinskoye deposit, Karelia, Russia), diatomite (Kamyshlovskoye deposit, Sverdlovsk region, Russia) and milled window glass (table 1).

All these materials are wide spread and very cheap in comparison with popular for silica nanopowder synthesis chemical precursors. Particle size range of used materials was not more than 2 mm.

Table 1. Content of SiO_2 in raw materials

#	Raw material	SiO_2 (wt. %)
1	Quartzite	99
2	Diatomite	80
3	Glass	75

Plasma synthesis of SiO_2 nanoparticles is carried out in experimental atmospheric pressure plasma reactor on the basis of DC arc plasma generator. The process of synthesis is based on processes of melting, evaporation and sublimation of raw material and next silica nanoparticles condensation from gaseous phase.

To evaluate quality of produced silica nanopowders all samples were compared with commercial ones: "Sky Spring" - nanosilica obtained from Sky Spring Nanomaterials, Inc., Houston- Texas, USA and "Tarcosil" - nanosilica obtained by electron beam way in Novosibirsk, Russia [5].

Different kinds of analyses (PSA, SEM, BET, EDX, XPS) were carried out to get information about sizes, morphology, chemical bonds and composition which influence the properties of material.

3. Results and discussion

Results of laser diffractometry (table 2) show strong agglomeration of silica nanoparticles in each test probe. An ultrasonic bath was used to prevent this factor. Diameters after ultrasonic treatment are ten times smaller but it was impossible to destroy agglomerates of particles during particle size analyses.

Table 2. PSA results

#	Raw material	Volume (%)	Diameter (μm)	
			As is	*Ultrasonic bath*
1.	Quartzite	10	9.0	0.4
		50	75.8	2.1
		90	338.2	42.4
2.	Diatomite	10	10.7	1.7
		50	100.3	10.9
		90	507.9	42.6
3.	Glass	10	41.3	1.5
		50	390.9	23.5
		90	783.9	70.1
4.	Tarcosil	10	-	9.0
		50	-	26.5
		90	-	74.0
5.	Sky Spring	10	-	4.8
		50	-	12.2
		90	-	32.2

Thus sizes of nanoparticles agglomerates from quartzite and diatomite are 42.4 and 42.6 μm which is close to sample "Sky Spring" with size 32.2 μm. Sizes of nanoparticles aggregates from glass (70.1 μm) were close to sample "Tarcosil" (74 μm).

Figure 1 illustrates the morphology and sizes of plasma synthesized nanopowders in comparison with commercial ones obtained after Scanning Electron Microscopy.

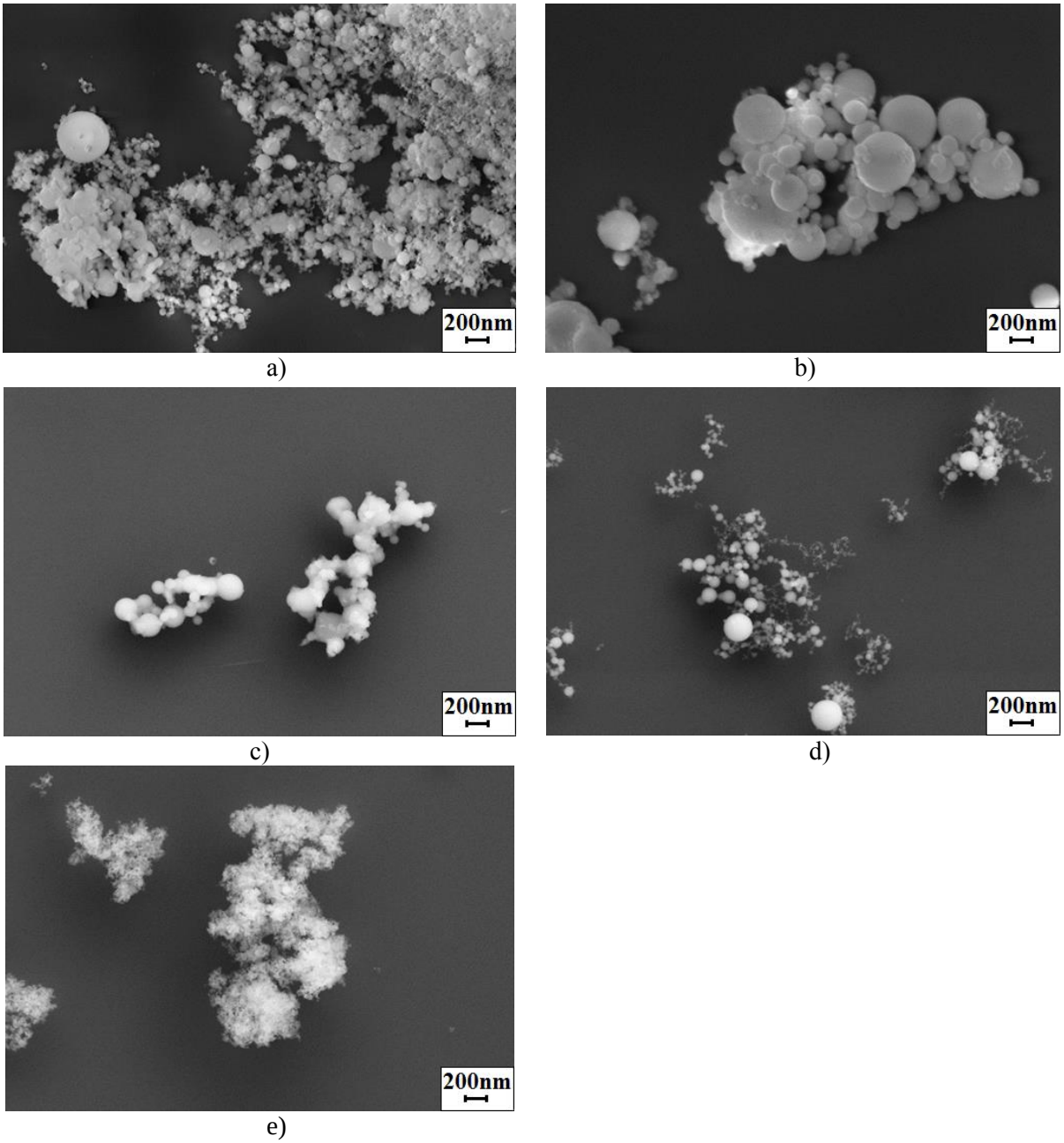

Figure 1. SEM images of nanopowders samples: a – Nanopowder from diatomite, b – nanopowder from quartzite, c – nanopowder from glass, d – Tarcosil, e – Sky Spring.

Investigations have shown that the nanoparticles have a spherical shape, polydisperse and agglomerated. The particle sizes in a range of 10-500 nm. The best result among synthesized nanopowders has the sample from diatomite: it has sizes similar to commercial nanopowders ones. The sample from quartzite has the biggest size among all nanopowders. Sample "Sky Spring" has the smallest dispersion in a range of 10-30 nm.

BET particle diameters (table 3) were calculated based on the measured surface area assuming monodisperse particles [13]. In order to calculate the equivalent diameter, the equation (1) was used:

$$D = \frac{6 \cdot 10^6}{\rho \cdot SSA} \quad (1)$$

Where ρ is bulk density of silica (2200 kg/m3), SSA is a specific surface area in m2/g. and D is the diameter of nanoparticles in nanometers. Plasma synthesized nanopowders were compared with only one commercial nanopowder (Tarcosil) in this analysis because it has comparable particle sizes.

Table 3. Specific surface areas and calculated BET diameters of silica nanoparticles

#	Raw material	SSA ($m^2 g^{-1}$)	Bulk density (g cm^{-3})	BET diameter (nm) (calculated)
1.	Quartzite	11.2	2,20	244
2.	Diatomite	32.2		85
3.	Glass	6.2		440
4.	Tarcosil	31.1		88

Thus SSA of diatomite nanoparticles (32.2 $m^2 g^{-1}$) is close to SSA of Tarcosil nanoparticles (31.1 $m^2 g^{-1}$). Calculated BET diameters were 85 nm and 88 nm correspondingly. Samples from quartzite and glass have smaller SSA (11.2 $m^2 g^{-1}$ and 6.2 $m^2 g^{-1}$) and are consequently bigger BET diameters of particles (244 nm and 440 nm).

Figure 2 demonstrates the elemental composition of nanopowders samples.

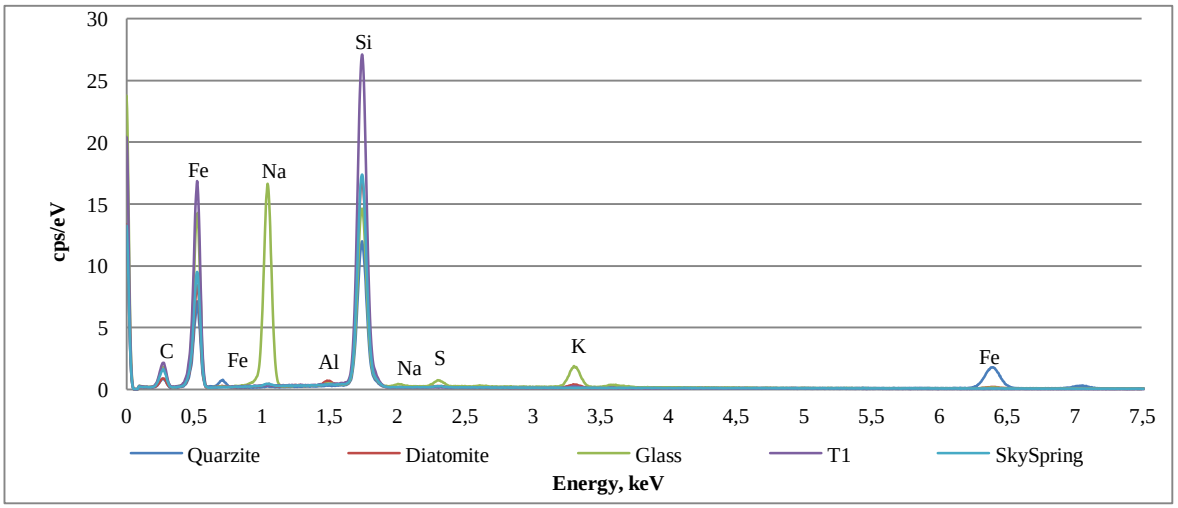

Figure 2. Elemental distribution pattern (EDX).

The pattern of distribution shows that the product contains predominantly the sublimation silicon and oxygen, which indicates representation nanopowder as silicon oxide. There are some impurities such as C, Fe, Na, K in nanosilica samples because a little amount of this element was in raw materials. The presence of carbon was due to the use of graphite as one of the electrodes in plasma system.

Results of XPS (Table 4) show quantitative evaluation of elements content in samples of nanopowders.

Table 4. Elemental composition of silica nanopowders (XPS)

№	Raw material	Element's content, at. %					
		O	Si	C	Fe	Na	K
1.	Quartzite	55.67	31.29	8.85	2.92	1.26	-
2.	Diatomite	59.78	31.15	6.63	0.94	0.81	0.70
3.	Glass	48.18	16.02	16.94	-	17.12	1.74
4.	T1	58.90	32.71	8.38	-	-	-
5.	Sky Spring	61.63	33.67	4.23	-	0.48	-

The highest level of impurities was found in the sample of nanosilica synthesized from milled glass due to its composition.

Conclusion

The possibility to obtain silica nanopowders from solid raw materials by DC arc plasma was shown. All produced samples of nanoparticles have a spherical shape, broad distribution of particle sizes and are strongly agglomerated. There are some impurities (C, Fe, Na, K) in synthesized nanopowders due to its presence in raw materials. Nanopowder from diatomite has nearly the same properties like commercial nanopowders.

Results of the investigation of plasma synthesized silica nanopowders show the possibility of their application in a building sector like additive to materials.

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