

Sonochemical Synthesis of the Silica Particles from SiC in Water Solution

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The paper presents a new method for producing nanosized SiO₂ particles by a sonochemical reaction in a mixture of SiC in distilled water. FTIR spectra exhibit absorption peaks characteristic of different oscillation modes in amorphous SiO_x. Optical transmission spectra demonstrate three characteristics components with energies of 2.9, 3.6 and 4.1 eV, which correspond to non-bridging oxygen hole centers and peroxide radicals ≡Si-O-O•; band at 3.6 eV realized in a chain ≡Si-O-O-Si≡ and point defects in ≡Si-O-Si-O-Si≡, respectively.

Keywords: Sonochemical reaction, Silicon dioxide, FTIR spectroscopy.

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1. INTRODUCTION

Metal oxides play an important role in various fields of physics, chemistry, and materials science. Metal oxides are used for producing sensors, piezoelectric elements, batteries, as well as for surface coating and passivation and catalysis [1]. As a result, transition metal oxides have found wide application in magnetoelectronics, photo- and solar cells, gas sensors, etc.

Silicon dioxide (silica) is the subject of intense study due to its physical and chemical properties. Silica exists in a variety of crystalline forms: quartz, cristobalite, tridymite, stishovite and coecite are the most studied, however, amorphous silica is the most common one. The properties of silica vary depending on its purity, shape, size, particle size distribution, formed transitions [2-9].

Silica can be obtained by different methods: sol-gel [10], deposition from the liquid phase [11], sputtering and deposition from the gas phase [12]. The synthesis of crystalline and homogeneous material for use in microelectronics, optical, electrical fields is of particular interest. The sol-gel method showed the synthesis flexibility, as well as the possibility of producing different configurations, such as monolithic blocks, coatings, fibers and films for application in optical devices. The metal salt is subjected to hydrolysis and a polycondensation reaction with the formation of a gel-like colloidal suspension consisting of a solvent and the metal oxide solid phase, whose morphology can be varied from discrete particles to continuous polymer networks [13]. The advantages of the sol-gel method include the ability to control the particle size and dispersion at low temperatures.

Nanosized silica particles with high specific surface were manufactured using a solution of sodium silicate in water and hydrogen chloride [14]. Most methods for the synthesis of nanosized silica and silica structures are based on tetraethylorthosilicate (TEOS) hydrolysis [15]. In the general case, the production of silica from TEOS consists of two steps: the hydrolysis of TEOS with the formation of silicic acids and the condensation as a result of the decomposition of silicic acids. The silica synthesis can be represented in the following form [16, 17]:



Sonochemical synthesis is one of the methods for producing low-dimensional semiconductor crystals. The phenomenon of cavitation is the basis of the sonochemical synthesis method of semiconductor crystals. Temperatures of 5000 K and pressures of 1000 atm can be reached when a "hot bubble" is collapsed [18-21].

The work is devoted to the first demonstration of the ability to produce SiO₂ particles by sonochemical method.

2. EXPERIMENTAL

Silicon carbide SiC (techn. > 98 % F150, grain size ~ 100 μm) and distilled water were used for silica synthesis in this work.

The synthesis was performed using a sonochemical reaction, for which 40 ml of distilled water was placed in the sonochemical reactor. After that, 1 g of silicon carbide was introduced into the sonochemical reactor. The radiation frequency (25-30 kHz) was adjusted and regulated to produce cavitation in a chemical solution. The sonochemical reaction proceeded for 120 min. Then, the reaction mixture was transferred from the sonochemical reactor. The silicon carbide precipitation, which did not enter into the sonochemical reaction, was separated from the silica solution by sedimentation. In the general case, the reaction of obtaining silica can be written as



Fig. 1 illustrates the mechanism of silica formation in this sonochemical reaction.

The chemical composition of the synthesized silica was analyzed using the infrared (IR) spectroscopy technique. IR absorption spectra were investigated in the range of 400-4000 cm⁻¹ using a Perkin-Elmer FTIR Spectrum BX II spectrometer. In this case, the synthesized silica was dried and studied in powder form. The optical absorption spectra were recorded on a MDR-23 monochromator and a hydrogen lamp as a light source. Optical absorption was recorded without additional processing of the obtained solution of silica particles in water.

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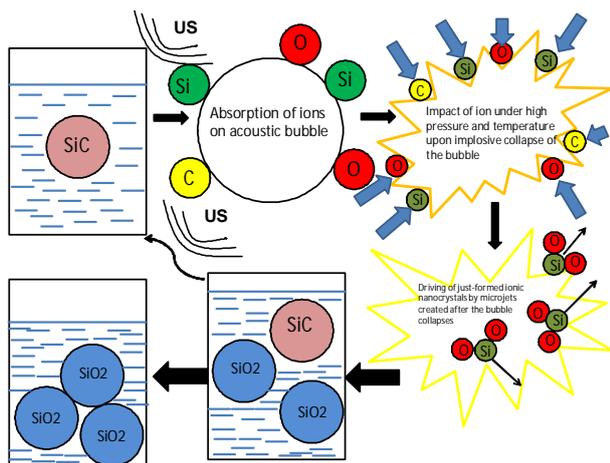


Fig. 1 – Growth mechanism of silica crystals in a sonochemical reaction

3. RESULTS AND DISCUSSION

A typical IR transmission spectrum of the synthesized silica is shown in Fig. 2. The detected FTIR peaks agree well with the results of previous studies of SiO_2 [10, 22-25], and also reveal the presence of silicon carbide residue, which did not enter into the chemical reaction.

A peak at 454 cm^{-1} with the vibration tension of the Si-O-Si bond was observed in amorphous SiO_x . The peaks at 822 cm^{-1} and 934 cm^{-1} are related, respectively, to oscillations in Si-O-Si and the vibration stretching of the Si-C bonds in the crystalline phase of silicon carbide.

The band in the spectral range of $1050\text{--}1300\text{ cm}^{-1}$ is more detailed in Fig. 3 (dotted line). Obviously, this band is formed by overlapping two vibrational modes, asymmetric stretching in Si-O-Si and stretching in Si-O-C with the corresponding peaks at 1094 cm^{-1} , 1146 cm^{-1} (solid lines in Fig. 3).

The band of the FTIR spectrum presented in Fig. 2 with the maximum at 2356 cm^{-1} correlates with stretching vibrations in silicon carbide. Peaks at 1405 cm^{-1} , 1632 cm^{-1} , 2922 cm^{-1} and 3442 cm^{-1} are associated with the vibrations of C=O, C-O, O-H, C-H_x and O-H, respectively. Obviously, these vibrational modes are formed due to the absorption of the corresponding molecular compounds on the surface of the synthesized sample. In Fig. 4 we schematically show the structure of the synthesized sample with the indicated absorbed compounds. Moreover, Fig. 4 illustrates the corresponding values of the peak maxima represented in the FTIR spectrum in Fig. 2 and indicates a complex, which forms a certain vibrational mode. Thus, the data of Fig. 2-Fig. 4 confirm the silica formation in the sonochemical synthesis proposed in this work.

According to the literature, the band gap for silicon carbide is 2.3-2.4 eV (3C-SiC polytype) [26], 3.0-3.3 eV (4H-SiC and 2H-SiC) [27, 28], 3.0 eV (6H-SiC) [29]. The silica band gap is equal to 7.8-9 eV [30, 31]. However, the absorption peaks in the range of 1.6-7.7 eV [32-34] are also manifested in silica that is realized on account of different complexes of bulk and surface defects. Moreover, the dependence of the position of indicated peaks on the particle size is observed for nanosized silica [33].

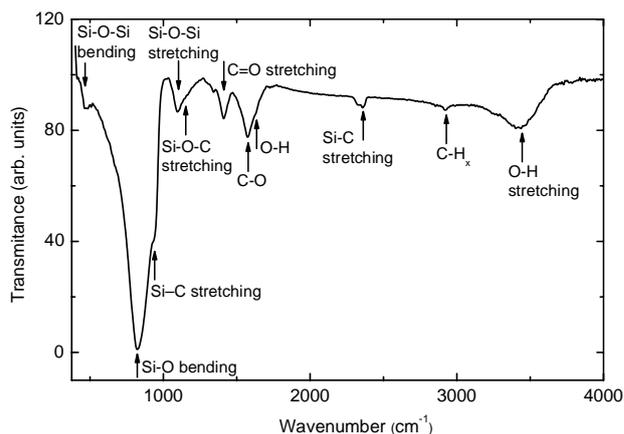


Fig. 2 – IR transmission spectrum of the sonochemically synthesized silica sample

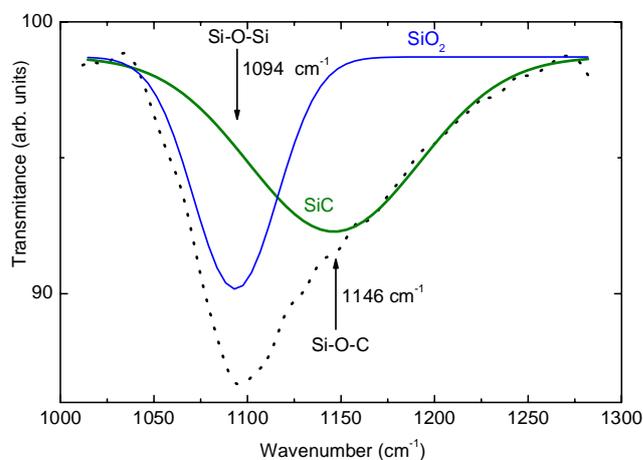


Fig. 3 – Separation of the vibrational bands of silica and silicon carbide (lines) from the fragment of the FTIR spectrum in Fig. 2 (dotted lines)

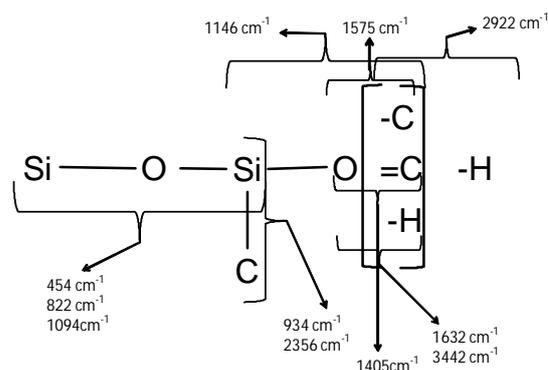


Fig. 4 – Schematic representation of the synthesized SiO_2 sample with the adsorbed compounds and the corresponding vibrational modes

Fig. 5 shows the optical transmission spectrum of the synthesized sample in the range of 4.5-2 eV. It is seen that three main bands at 2.9 eV, 3.6 eV and 4.1 eV are observed. The band at 4.1 eV is associated with the non-bridging oxygen hole centers [35], as well as with the peroxide radicals $\equiv\text{Si-O-O}\cdot$ [36]. The band at 3.6 eV can be compared with the known band realized in the chain

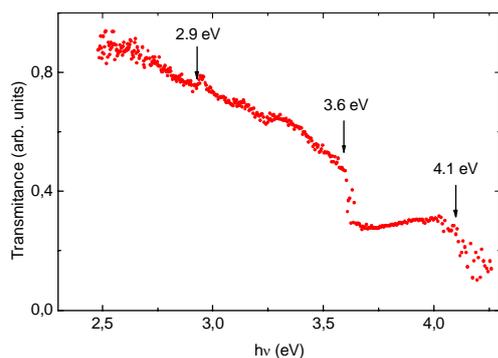


Fig. 5 – Optical transmission spectrum of the synthesized silica in water solution

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$\equiv\text{Si-O-O-Si}\equiv$ [32]. The band at 2.9 eV can be related to point defects in dicoordinated silicon lone pair $\equiv\text{Si-O-Si-O-Si}\equiv$ [33, 35]. We note that the photoluminescence spectra of nanosized silica in the visible range are formed in these bands due to the absorption [35].

Thus, the possibility of producing SiO_2 particles by the sonochemical method using silicon carbide as a precursor is shown for the first time in the work. The molecular structure of synthesized silica is confirmed by the obtained IR absorption spectrum. The most intense vibration modes correspond to Si-O-Si vibrations in amorphous SiO_x , Si-O-Si and Si-C in the crystalline phase of silicon carbide, respectively, by asymmetric stretching in Si-O-Si and stretching in Si-O-C.