SYNTHESIS OF CARBON NANOSTRUCTURES IN COMPOSITES-MATRICES BASED ON FUMED SILICA

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Introduction

Numerous methods for producing nanoparticles in the form of inclusions in porous matrices have been developed to date. An advantage of nanoparticle synthesis in chemically inert matrices is that the matrix prevents aggregation of nanoparticles formed and protects them from external actions. Direct formation in the bulk of the matrix makes it possible to control the parameters of nanoparticles at the stage of their formation. By choosing matrices with different shape of structural voids, one can synthesize nanostructures of different morphology and anisotropy [1].

The process for producing porous amorphous silica (mesoporous molecular sieves) by template synthesis involves three stages: gel synthesis, drying and removal of the template. Materials based on mesoporous silica are used as catalyst supports, membranes, selective adsorbents, sensor materials, and matrices for producing a great number of functional composite materials [1].

In recent years, the "nanotectonics" method, which consists in producing an ordered structure of dispersions of spherical micro- and nanoparticles, has been employed to make porous inorganic materials with a system of ordered pores. Depending on the mode of interaction between particles, porous materials are produced, which are a combination of two-dimensional and three-dimensional ordered assemblies of particles, mesoporous structures, and aggregates of particles of specific shape [2].

The aim of this work was to synthesize pyrocarbon from polystyrene nanostructures in composites-matrices based on fumed silica.

In contrast to the matrix method for the synthesis of mesoporous silica, the composites-matrices produced are porous structures of nonporous particles of amorphous SiO_2. The self-assembly of nanoparticles is the keystone of the proposed method for the matrix synthesis of carbon nanoparticles.

Results and Discussion

The precursor for the preparation of pyrocarbon was polystyrene (PS), which was carbonized at 750 ^{o}C under an argon atmosphere. Solutions of polystyrene in ethyl acetate were used as a dispersion medium in the preparation of composite from fumed silica (particle size $\leq$80 nm) or from it and glass microspheres (particle size 10 – 40 microns). To investigate by transmission electron microscopy (TEM) the size and shape of the carbon structures obtained, their dispersions in acetone were used after etching away silica and glass spheres with a mixture of concentrated sulfuric and hydrofluoric acids.

In polymer solutions there is an equilibrium between associated and individual macromolecules. Dilute solution consists of individual macromolecules, between which there is practically no intermolecular interaction. Increasing the polymer concentration leads to an increase in the size of macromolecular aggregates and their number in unit volume. In concentrated polymer solutions, the size of aggregates decreases and approaches the size of undisturbed macromolecules. In Ref [3], the dimensions and concentration of macromolecular aggregates for solutions of PS in ethyl acetate have been determined.

The process of formation of the composite-matrix from SiO_2 particles is influenced by polystyrene macromolecules. The composite is formed by interaction between SiO_2 particles (aggregation) and interaction of these particles with glass spheres and PS macromolecules. This leads to the formation of complex-shaped structures, which are connected by a system of nonvalent interactions and consist of polymeric-inorganic blocks of a great number of particles.

Figure 1 shows carbon nanostructures obtained by PS carbonization in a composite of only silica particles. At the PS concentration used, polymer is localised on the surface of only one SiO_2 particle: on carbonization, mainly carbon structures as segmented spheres of up to 60 nm size are formed. Larger structures broke apparently down when samples were prepared to study them by TEM. Adsorption of macromolecules between SiO_2 aggregates and particles leads to the formation of an additional number of ovoid and conical carbon particles.

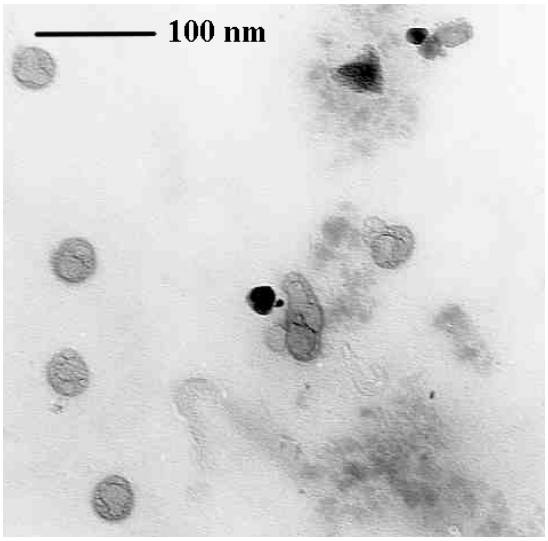

Fig.1. Transmission electron micrograph of carbon nanostructures obtained by polystyrene carbonization in a silica composite-matrix. The PS concentration was 5 g/ 100 ml.

In composites of silica and glass spheres, pores of maximum size are formed between glass spheres and aggregated SiO_2 structures. PS is adsorbed mainly on the silica surface in comparison with glass spheres [4]. It may be assumed that in the case of large size of aggregates of PS macromolecules in the solution [3], macromolecules will be adsorbed on silica particles that formed in adsorption layers through interaction between macromolecules. When PS is carbonized in such composites, carbon flakes of 15 – 150 nm size are formed (Figure 2).

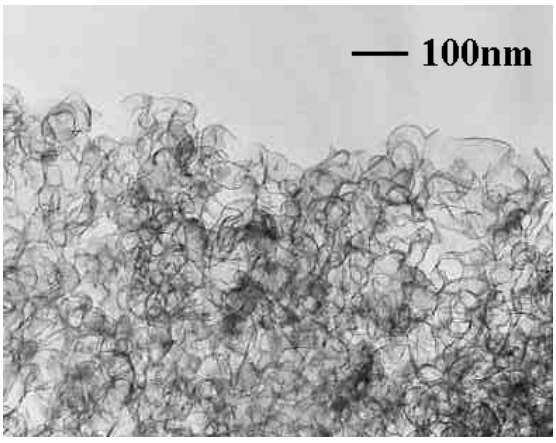

Fig.2. Transmission electron micrograph of carbon nanostructures obtained by polystyrene carbonization in a composite-matrix of silica and glass spheres. The PS concentration was 1.5 g/ 100 ml.

Increasing the PS concentration results in complete coverage of the surface of SiO_2 particles with polymer layer. Because of steric effects, the adsorbed macromolecules are forced to retain the conformation that they had in the solution. On carbonization, segmented spheres, which are analogous to those shown in figure 1, are formed in such composites.

Conclusions

Carbon nanostructures have been obtained by matrix synthesis by forming a composite-matrix in the presence of carbonization precursor -– polystyrene.

The dependence of the size and shape of carbon nanostructures formed on the composition of the composite-matrix and precursor concentration has been established.

References

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