Electric-Arc Plasma Installation and Method for Preparing Nanodispersed Carbon Structures

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ABSTRACT

An electric-arc plasma installation operated in the "hidden" anode arrngement is constructed and used for the pereparation of carbon nanostructures. A device and a method were developed to produce nanomaterials in the form of nano-particles that are stable, polymorphic, head modified crystal structures of any type of elecrtically conducting materials, in particular of graphite, and possess unique physical, chemical and mechanical properties. The operating technological parameters of the contracted plasma arc were deternined in view of obtaining nanosised carbon structures withe given shape (nano-tubes, fullerenes, clusters, disc-like shapes, leaf-shaped etc.), size and structure.

The method, the divice and the nanomaterials produced can find application in the plasma-chemical industry, the nanotechnologies, the space technologies, the medicine and for fabrication of composite materials.

***Keywords*:** thermal plasma method, carbon nanostructures, properties

1 INTRODUCTION

The high energy parameters of low-temperature plasma (LTP), the high rates of the vaporization and condensation processes, combined with the possibilities for automation, optimization and modeling of the plasma-chemical processes, determine the practical expediency of employing LTP for nano-dispersed powders (NDPs) preparation. Thanks to the exceptional plasma properties (high temperature and energy density, presence of a large number of charged particles, etc.) and to the specific gas-dynamic and thermo-physical conditions existing in the plasma-chemical reactor (PCR), all physical and chemical processes involved in the plasma treatment of any initial material (heating, vaporization, thermal dissociation, chemical interactions, nucleation and condensed-phase particle growth) take place at very high rates and, therefore, for a very short time (10^{-2} – 10^{-3} s). As a result, the desired products obtained are in most cases characterized by a very small particle size (1 – 100 nm) and high number of defects in the crystal. In many cases (high-melting point nitrides, oxides, carbides, etc.), the plasma-chemical (PC) treatment is the only possible technique for preparation of NDPs [1, 2].

At present, carbon nanostructures are regarded as artificially composed structures with nanometer size. Their properties are the subject of both theoretical and experimental investigations; and are expected to find a very wide range of possible applications. The history of carbon nanostructures begins in 1985, when the Buckminster fullerene C60 was discovered by Kroto [3]. Since then, the number of discovered structures has been rapidly increasing. Examples of them are: the nanotubes discovered by Ijima [4], the family of fullerenes C70, C76, C84, C60 in a crystalline form, carbon nanocones, carbon nanohorns, nanoscale carbon toroidal structures and helicoidal tubes, etc. These carbon structures could be single-walled or multi-walled; they may have zero, positive or even negative Gaussian curvature (Schwarzites). Recently, a few types of similar non-carbon structures were discovered, such as boron nitride nanotubes, molybdenum disulfide or tungsten disulfide structures and even silicon nanotubes.

The aim of the study reported here is to use a modified technique of interaction between the material treated and a contacted plasma arc; the latter is transferred directly on a fixed or moving graphite electrode in the "hidden" anode arrangement thereby making it possible to achieve temperatures about 6 000 K in and above the interaction zone. The electrode material is thus vaporized and, following quenching and fixing, nanosized carbon structures are obtained.

2 EXPERIMENTAL

A schematic of a plasma-chemical installation for production of carbon nanostructures operating under the "buried" anode arrangement is shown in Figs. 1 and 2 [5]. The plasma arc (38) generated by the plasma torch (1) (see Fig. 1) has mass-averaged temperature exceeding $10x10^3$ K.

The arc is strongly contracted, intensive and with high speed of motion of the current-carrying plasma stream. The plasma arc is transferred directly on a cylindrical graphite electrode (anode - 8) in the "buried" anode arrangement. The plasma arc and the plasma stream envelops entirely the anode contact area formed (40). As a result of this frontal attack with high heat power density (about 2 kW/mm^2), a temperature of about 6 000 K is reached in "hidden" anode contact area and above it. This results in intensive explosive vaporization of the graphite, which sublimates into a solid aerosol phase. The vaporization process takes place continuously, as the vaporized material is replaced be new one by means of moving the electrode in a direction

opposite to the plasma stream. The electrode diameter is smaller than that of the positive column of the contracted plasma arc formed by the plasma torch nozzle. Thus, complete "burying" is ensured of the anode contact area within the moving plasma stream and a temperature of 6 000 K is permanently maintained in this area throughout the entire vaporization cycle.

A part of the plasma arc and the plasma stream after the contact area move into a closed space within the chamber. It is formed by two hollow open cones located within a closed space (41), the latter being connected through holes to another multi-chamber space used for collection of the nanomateriales produced. The open cones are coaxial with respect to one another and to the electrode (8); their walls, whose geometry can be varied, are formed by a moving fluid. The walls quench, mix and support the saturated plasma-aerosol carbon phase produced through vaporization of the electrode material. This phase is carried by the plasma stream, which leads to the initiation of homogeneous crystallization of carbon nuclei followed by quenching at a given moment. The latter interrupts the crystallization process and ensures the formation of heat-modified stable polymorphic nanosized crystalline carbon particles. If the graphite electrode is replaced by an electrode of another electrically conducting material, one will obtain nanoparticles of that material.

Throughout the vaporization cycle, the plasma arc remains with constant length and very narrow temperature profile. Thus, the installation allows one to perform continuous and complete transformation of the entire initial material into a plasma-aerosol phase.

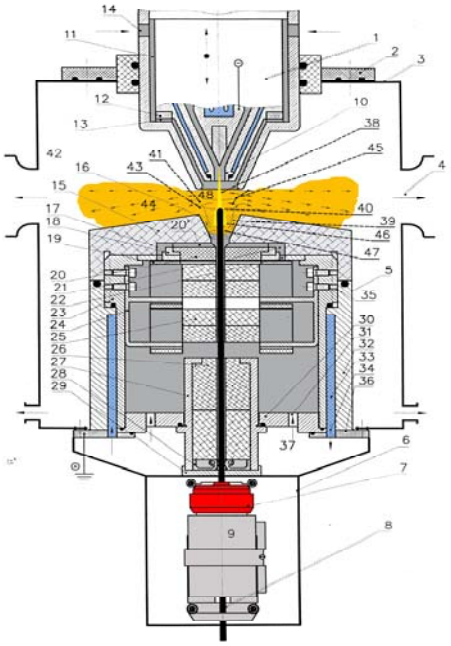

Figure 1: Schematic of plasma unit for production of nanosized carbon structures

1 - water-cooled elongated cylindrical three-body plasma torch; 2 - insulating air-tight coupler; 3 - chamber; 4 - holes for removing the products; 5 - two-body vaporizer; 6 - fixed electric power supply; 7 - rotating planetary head; 8 - electrically conducting anode of the plasma torch; 9 - electrical motor; 10 - plasma torch nozzle; 11 - gap; 12 - stream twisting device; 13 - tangential channels; 14 - holes for feeding quenching fluid; 15 - protective lid; 16 - tapered hole; 17 - cylindrical hole; 18 - holder; 19 - supporting metal disk; 20 - stream twisting device; 20' - tangential channels in the stream twisting device; 21 - gap; 22 - holes; 23 - internal body; 24 - spring holders; 25 - current feeding brushes; 26 - current feeding cylindrical graphite bushings; 27 - metal cylinder; 28 - silicon rubber seal; 29 - cap; 30 - metal bottom; 31 - holes for quenching fluid; 32 - outside housing; 33 - cooling gap; 34 - holes; 35 - holes for cooling liquid; 36 - flange; 37 - fixing plate; 38 - plasma arc; 39 - saturated plasma-gas-aerosol phase; 40 - anode contact area; 41 - volume enclosed by the moving fluid walls; 42 - body; 43 - inter-electrode gap; 44 - vapor-gas mixture; 45 - moving conical fluid wall; 46 - mixed gas-plasma stream; 47 - lower open conical space; 48 - upper open conical space;

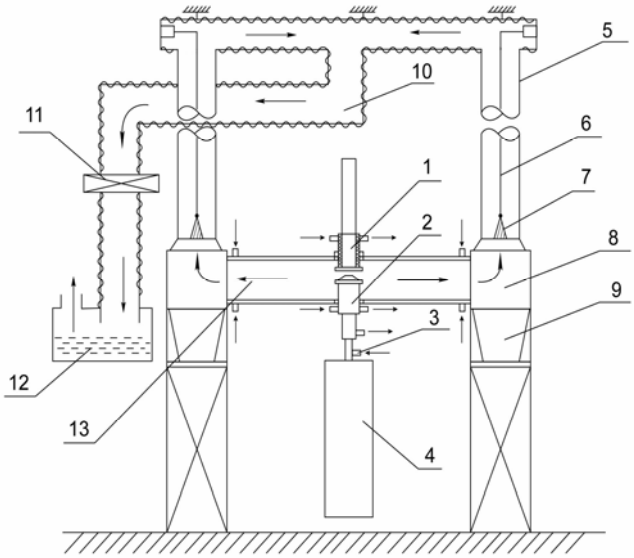

Figure 2: Schematic of the plasma installation for production of nanosized carbon structures

1 - plasma torch coupled to a quenching device; 2 - vaporizer coupled to an electric power supply; 3 - electric power supply; 4 - magazine-type electrode-feeding device; 5 - electrostatic filter; 6 - electrostatic filter cathode; 7 - counterweight of the electrostatic filter cathode; 8 - chamber-electrostatic filter coupler; 9 - bunker collecting the nanodispersed material; 10 - pipes for the stream after the electrostatic filter; 11 - suction fan; 12 - water filter; 13 - quenching chamber.

3 RESULTS AND DISCUSSION

The nanostructures produced were studied by a Hitachi S570 TEM at magnification as denoted on each microphotograph, depending on the size of the structures and the preparatory techniques used. The TEM images show spheres of very small size (about 10 nm) (Figures $3^a \div 3^s$), as well as tubular structures clogged at both ends by spherical formations.

The samples thus observed can be described as follows: the presence is seen of crushed graphite (Figs. 3^f, 3^l, 3^m), of spherical formations of various size (Figures. 3^a, 3^g, 3^o), of clusters (Figures. 3^k, 3^n), of elongated structures (Figures. 3^h, 3^j, 3^p) resembling tubes or threads (Figures. 3^h, 3^j, 3^p). Some of the spherical formations have holes, so that one can assume that they are hollow. The TEM images show spheres of very small size (about 10 nm), as well as tubular structures clogged at both ends by spherical formations (Figures. 3^j, 3^k, 3^p).

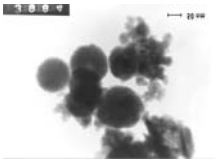

Figure 3^a

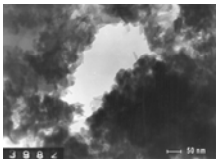

Figure 3^b

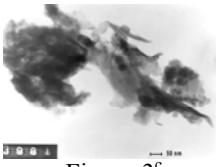

Figure 3^c

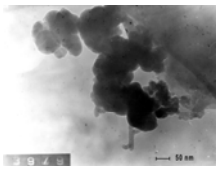

Figure 3^d

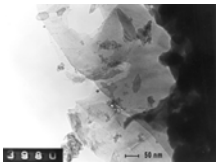

Figure 3^e

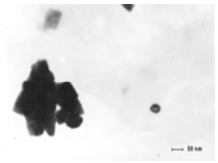

Figure 3^f

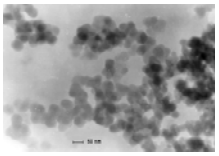

Figure 3^g

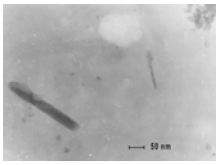

Figure 3^h

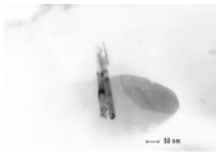

Figure 3^i

Figure 3^j

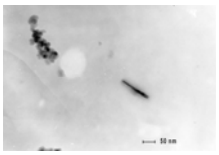

Figure 3^k

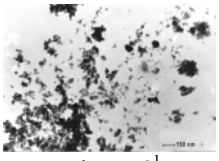

Figure 3^l

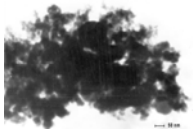

Figure 3[m]

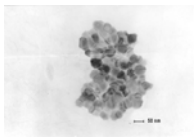

Figure 3[n]

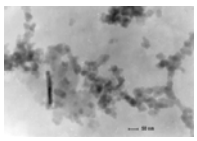

Figure 3[o]

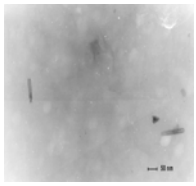

Figure 3[p]

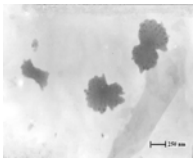

Figure 3[q]

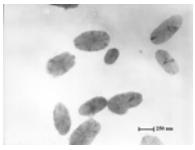

Figure 3[r]

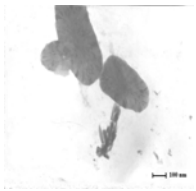

Figure 3[s]

Figure 3: TEM images of synthesized nanodispersed carbon structures

The TEM images of a sample extracted from water by using toluene show particles of elongated or disc-like shape. Some of them resemble leaves with embedded small black particles of various geometry. One can also see conglomerates of complex shape consisting of spherical and elongated particles. The TEM image at magnification of $4x10^3$ shows that their size is about 50 nm. At magnifications of 180 000 - 200 000, one can see that the discs are of variable density, decreasing to the periphery with a sharply defined borderline; the discs have sizes of about 10 - 25 nm. Spherical particles are also present in this image, which are also of variable density and sizes of about 10 - 25 nm. One can further observe tubular structures with channels in the middle. The outer diameter of the tubes is 10 nm, the inner, 2 nm, and the overall length is 50 nm.

At a magnification of $2x10^5$, one can see a mesh-like structure containing spherical dot formations with sizes of about 5 - 10 nm. Needle-like and triangular formations are also present that exhibit multi-layered structures. The results of the X-ray phase analysis show two clearly seen lines (d = 2,81 Å and d = 3,00 Å), which describe the carbon nanostructures.

4 CONCLUSIONS

1. The application of a technique employing a contracted plasma arc transferred directly on a moving cylindrical graphite electrode (anode) in a "buried" anode arrangement allowed the authors to produce nanosized carbon structures of various size and texture. The high energy parameters of the contracted plasma arc determine the efficiency of the plasma process in what concerns the graphite material vaporization.
2. The operating technological parameters of the contracted plasma arc were determined in view of obtaining nanosized carbon structures with given shape, size and structure.
3. Collection of the nanosized carbon structures by means of a quenching water cone is efficient and environmentally appropriate.

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