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Complex particles produced from graphite powder by acoustic cavitation in water

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Abstract

Spaghetti-like structures made of fibers whose diameter are of about $1-2 \mu m$, straight structures from about $10-20 \mu m$ and optically semitransparent particles, with linear dimension up to about 0.3 mm, were obtained from fine graphite powder in water subjected for 20 h to acoustic cavitation at a frequency of 19.5 kHz.

A picture of spaghetti-like structures obtained by scanning electron microscopy is reported. From X-ray diffraction pattern, it was found that the semitransparent precipitates have an amorphous nature. © 2002 Elsevier Science B.V. All rights reserved.

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In recent years acoustic cavitation received new deal of attention mainly because of the discovery of two effects, which show that both practical applications and fundamental mechanisms of matter interaction related to this phenomenon, carry on reserving surprising peculiarities. Indeed, while Suslick and co-workers discovered the possibility of the production of non-crystalline metal phases [1], opening up new technological opportunities and new theoretical debates, Barber and Putterman observed the picosecond light emission of sonoluminescence [2].

Nowadays the new possibilities opened up by Suslick ten years ago seem to introduce in a completely unexplored field. There is the perspective on a technological use of non-crystalline powders so produced. This possibility can justify a new effort in order to solve some problems, such as to control quantity, purity and macroscopic form of the produced nano-structured materials. These difficulties explain the non-fulfillment of the hopes initially born with such technique. In fact, until now, besides Iron, produced by Suslick and his coworkers, only magnetite [3–5], cobalt [6], nickel [7,8], palladium [9–11], chromium [11], silver, copper and lead selenides [12], were synthesized. In addition it is not yet clarified the exact chemical reaction, which produces the new compounds and structures, and its possible relationship with the mechanism producing the observed strong damage of solid surfaces [13,14]. In our opinion, the modest knowledge of physics and chemistry at the temperature (5000–10000 K) and pressure (up to 1 GPa), existing in the imploding bubbles, explains the confusion present in the field.

As a consequence of this situation it is only the flair to suggest making a given experiment.

Tubules of carbon with various diameters, up to nano-tubes and polyhedral particles can be generated in arc discharge between graphitic electrode [15–17], where high electric field and high temperature are present, so we thought that powder of graphite, subjected to cavitation, could produce something of similar to the electrical arc-discharge products, since at the "extreme conditions" due to cavitation, microplasma highly charged is generated [18,19].

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With this letter, we communicate that powder of ultra-pure graphite, dispersed in pure water, subjected for a suitable time to strong cavitation, generate several precipitates: (i) *spaghetti-like* structures made of fibers whose diameter is from about 1–2 μ m; (ii) elongated structures with diameter up to about 10 μ m; (iii) optically transparent or semitransparent particles, very easily visible to the naked eye for color and dimension, whose size is up to 0.03 mm³.

These precipitates have been obtained using 0.6 g of graphite of nominal purity 99.997% (Goodfellow Metals). Its typical analysis was: Al 0.2, B 1, Ca 0.05, Cu 0.01, Fe 0.2, Mg 0.05, Na 0.02, Si 1 ppm. We bought rods of C, length 100 mm and having a diameter of 13.0 mm. We were able to obtain a powder whose maximum particle size was less than 50 µm, by rubbing two graphite rods each against the other. Industrial powders, like those supplied by Goodfellow Metals for instance, have too many particles with a size of about 200 µm. The fine powder we obtained was then dispersed in 200 ml of deionized and bi-distilled water contained in a fused silica bottle made. The bottle was hermetically sealed directly to the horn of the vibrator. Water and powder were then subjected to acoustic cavitation at a frequency of 19.5 kHz, for 20 h at a constant temperature of 0 °C. The applied power transferred to the liquid was of about 200 W/cm².

When the graphite powder is originally dispersed in water, the smallest particles stay afloat on the surface of the liquid, producing a very thin pellicle gray metallic colored, the greatest precipitate on the bottom of the container. The remaining particles are uniformly dispersed in the water bulk.

During 20 h of strong cavitative activity, no precipitate was observed. A dark powder precipitate started to appear a few hours after the end of the irradiation and the process ended after about 10 h, when the water got back to its initial transparency. To avoid any contamination due to the presence of external air, we left the container tightened to the horn for five days. In this period no alteration regarding the powder was observed as well as when we unscrewed the container from the horn. Only after about a month, when the container was left accurately closed, it was possible to observe many dark filiform structures, a dark, wool-like matter and many transparent or semitransparent particles.

The material produced was extracted from water and selected. The precipitate contained various components having different morphology and probably different chemical nature. In the present test the amount of material, we treated was too small for an accurate chemical analysis, therefore we confined ourselves to a microscopy study and some X-ray diffraction investigation.

The most part of the precipitate was composed by a black powder, reminiscent of the original graphite powder. An X-ray diffraction study of the semitrans-



Fig. 1. Spaghetti-like structure as seen by electron microscopy.

parent particles, performed using Mo K α radiation, suggested that they have a non-crystalline structure. The particle solubility in the acetone supports the hypothesis that they are produced by the polymerization of an organic substance. A detailed analysis of these particles will be reported as soon as possible.

In Fig. 1, obtained by electron microscopy, we show the image of the spaghetti-like structures. These fibers appear to have a quite complex morphology.

X-ray analysis of the graphite before treatment and of the black powder after cavitation, were made using a monochromatic Cu K α radiation. The diffraction pattern of the black precipitates after cavitation is much more complex than that of the original graphite and we leave a full analysis of each precipitate, to a forthcoming publication.

In conclusion, we have reported a new application of the cavitation technique, which could open new perspectives. We have shown that even starting from an elemental system, like pure graphite, it is possible to produce various different compounds with different atomic arrangements as well as shapes. It is clear that much more effort has to be devoted to the study of the present cavitation products in order to define the physical and chemical nature of the various precipitates. Nonetheless the present results introduce further development perspectives to the cavitation approach to stimulate new reaction processes.

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