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Dissipative structures in thin films of cavitation-activated soot suspensions

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Abstract. This paper presents analysis of optical images of films obtained by evaporation (293 K) of a thin layer of low-concentrated (1% wt.) aqueous soot suspensions in a Petri dish. Suspensions of wood, fullerene and diamond-containing (after detonation synthesis) soot were prepared using hydrodynamic dispersion in a cavitation mixer with a wedge-shaped cavitator with the rotor's angular velocity of rotation being 10000 rpm. The components of the hydrodynamic dispersion process are turbulent micro-mixing and bubble cavitation. As a result of intense turbulence, carbon suspensions assumed properties of non-equilibrium systems. The images of an optical microscope revealed self-organized dissipative micron-sized structures in the dried films being present there in the form of chain, branched and ring-shaped cluster fractals of various dimensions that look very similar to nanotubes. The self-assembling effect was identified for all the types of suspensions under study. The EPR method was used to study the change in the electronic structure of soot resulting from the exposure to high temperatures and pressures that occur when the cavitation bubbles collapse (cavitational activation). The dynamics of changes in dissipative structures in the process of film drying was monitored.

1. Introduction

The study of self-organization of carbon nanoscale systems is an important research field fostering development of modern nanotechnologies [1–3]. In this study the method of hydrodynamic dispersion of aqueous carbon black suspensions in the region of high velocities was used in order to obtain non-equilibrium states of carbon nanosystems. Fullerene, detonation diamond and wood soot were used. The study was focused on images in thin films of evaporating soot suspensions put in a Petri dish. Colloidal film evaporating from the glass surface is an open system with a variable mass. Such a system exchanges substance and energy with the environment. Self-organisation processes occur in the liquid layer, generating space-time dissipative carbon structures.

The components of the hydrodynamic dispersion technology are intensive turbulent micro-mixing and bubble cavitation. Dispersion of soot aggregates having a branched structure and a change in the electronic structure of soot globules (cavitation activation) occurs as a result of shock waves near collapsing cavitation microbubbles (CM) and cumulative microstructures emerging during asymmetric collapse of CM. When bubbles collapse in a local volume, near it and inside, high temperature fields (103–104 K) and pressures (≈ 1000 MPa) are formed. This is the area where various chemical reactions and phase transformations can occur [4].

2. Experimental techniques

It is known that energy-efficient hydrodynamic dispersion technology (grinding in a liquid medium) can be used for fine grinding of materials [4]. The dispersion of carbon black suspensions was carried out in a hydrodynamic rotary oscillator with a two-blade wedge-shaped impeller in supercavitation mode. The engine capacity is 1 kW, the volume of the working chamber is $3 \cdot 10^{-4}$ m^3, the angular velocity of the rotor is 10000 rpm. In the supercavitation mode the bubbles are localized in the tail part of the non-stationary supercaverns arising behind the rotor and a stable cavitation effect on the dispersible phase is attained without erosive destruction of the impeller.

The size distribution function and the average size of the carbon black particles were determined by electron microscopic images (JEOL JEM 2100 microscope). The study of the electronic properties of source and cavitation activated carbon blacks was performed on an SE / X-2544 EPR spectrometer at a frequency ν = 9 GHz, λ = 3 cm at 293 and 77 K. The structure of the dried carbon black suspension's film in a Petri dish was studied on an XSP-optical microscope 128H by OptiTech, connected to the computer (with magnifying power being equal 400x), and on a digital optical microscope HIROX KH-7700.

The elemental composition of the initial samples of wood soot was determined in wt.% using S-4 Pioneer X-ray fluorescence spectrometer by Bruker with accuracy exceeding 0.001% depending on the element.

The experiment was carried out on the following types of soot: wood, fullerene soot containing 11% of C_{60} (produced by Neo Tech Product, St. Petersburg, Russia), detonation nanodiamond soot obtained by detonation synthesis (Altai, Biysk, Russia). The composition of the soot samples is shown in table 1.

Table 1. Elemental composition of soots.

	Wood soot	Fullerene soot 11% C_{60}	Detonation diamond soot [6]		Wood soot	Fullerene soot 11% C_{60}	Detonation diamond soot [6]
C	94.580	99.92	82.28	Ca	0.1	0.0033	-
Na	0.1	0.0071	-	Cr	0.356	-	0.50
Mg	0.18	0.0016	-	Fe	0.11	0.0028	4.52
Al	0.382	0.0041	1.27	Co	0.373	-	1.92
Si	-	0.019	0.42	Ni	0.639	0.0018	-
P	-	0.0015	-	Cu	3.001	0.0039	0.61
S	-	0.013	4.33	Zn	0.0759	0.0028	-
Cl	-	0.022	-	O	-	-	4.23

For the experiment, there were prepared low-concentrated (1% wt.) aqueous suspensions of soot powder in distilled water. The choice of concentration was made judging from the condition of minimum coagulation of soot particles. The water suspension was processed for 60 s in the supercavitation mixer at a rotor speed of 10000 rpm. Then the suspensions were poured in a thin layer into Petri dishes and dried in air at 300 K. For measurements of EPR and EM dried powder in a Petri dish was used.

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3. Experimental results and discussion

3.1. Dispersion of soot aggregates

Electron microscopic images of the initial soot types are shown in figure 1a. These are aggregates in the form of branched chains consisting of soot globules, “stitched” among themselves. Aggregates are grouped into less durable agglomerates.

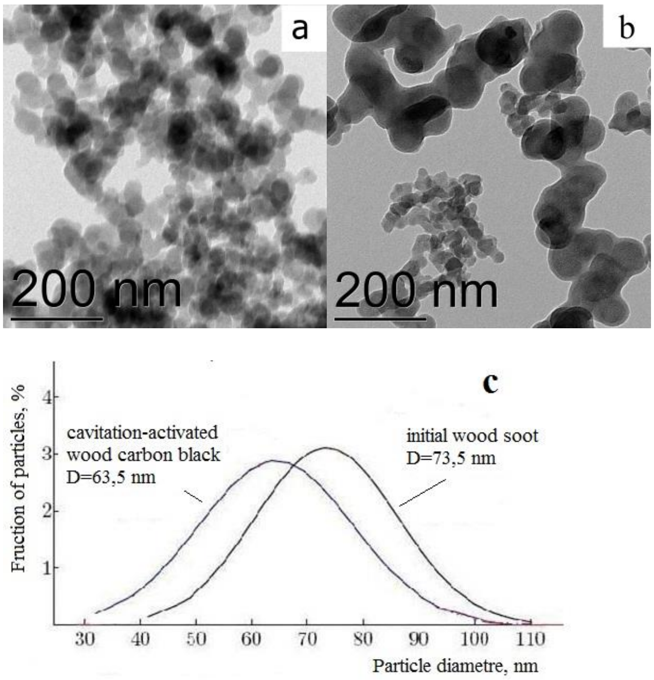
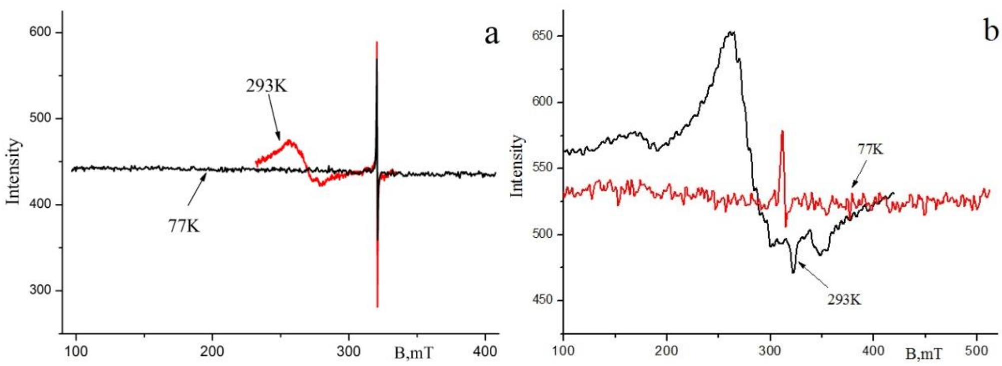


Figure 1. Electron microscopic images of (a) the original wood soot sample, (b) of the same sample after hydrodynamic dispersion and their particle density distribution function (c).

Figure 1b shows that, when dispersed, the average size of the globules decreases by approximately 15%. For the original wood soot, the globule size is 73.5 nm whereas for the activated one it is 63.4 nm, the distribution law is normal, the dispersion of the distribution curve is significant, which indicates large size dispersion (figure 1c). In a similar experiment with detonation diamond soot the average size of the globules after dispersion was equal to ≈ 47 nm [5].

3.2 ESR spectra of soot suspensions

To study changes in the electronic structure of soot particles during cavitation EPR spectra were analyzed (figure 2).



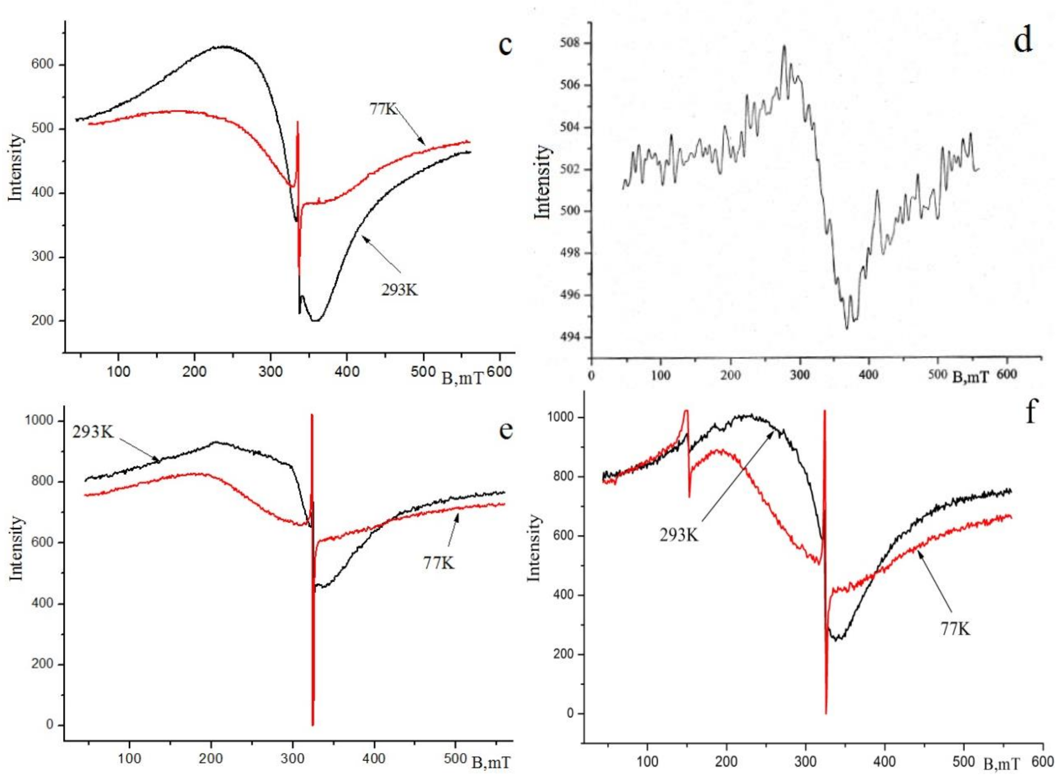


Figure 2. EPR spectra: (a) 11% C_{60} fullerene soot, (b) 11% C_{60} cavitation-activated fullerene soot, (c) detonation diamond soot, (d) cavitation-activated detonation diamond soot (77 K), (e) wood soot, (f) cavitation-activated wood soot.

3.3 Dissipative structures during evaporation of a thin film of soot suspension in a Petri dish
The images of soot films in a Petri dish were analyzed on an optical microscope (figure 3).

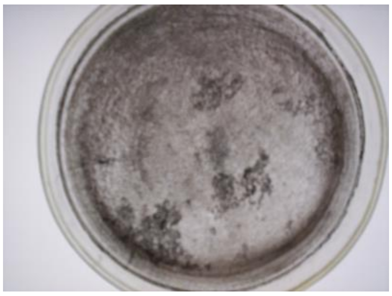


Figure 3. Soot film in a Petri dish.

A thin evaporating layer of soot suspension after hydrodynamic dispersion can be regarded as a non-equilibrium system with a large amount of stored energy generated in the course of cavitation effects. The images in an optical microscope (figure 4) in dried films revealed self-organized dissipative structures in the form of chain-, branched and ring-shaped cluster fractals of various dimensions, externally resembling nanotubes [7]. Self-assembly took place when there was a significant amount of impurities in the soot (table 1).

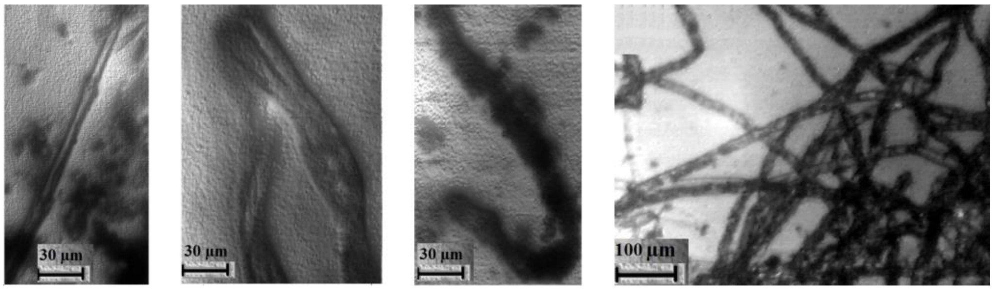


Figure 4. Optical images of dissipative structures in soot films (self-assembly).

Observation of the dynamics of fractals had been carrying out for 6 months. With the same experimental parameters (e.g. speed of rotation of the cavitator, duration of rotation), self-assembly of structures in wood soot films occurred 3–4 days later than in films of fullerene-containing and detonation diamond soot. In the films of fullerene and detonation diamond soot, linear, branched and ring-shaped fractals were observed. There were especially many of them in diamond films. In wood soot films the fractals were mostly linear, and were in smaller quantities.

The self-assembly process of dissipative structures had been taking place actively for 2 months before the period of their destruction began. However, 6 months after the start of evaporation of the sample of cavitation-activated detonation diamond soot, all types of fractals were present in the film. In wood soot films fractals disappear earlier and are more mobile. These features might be explained by the additional evaporation of free and bound water which are found in types of soot as a result of cavitation effects [8].

4. Conclusion

As a result of intense turbulence and bubble cavitation during hydrodynamic dispersion water soot suspensions based on wood, fullerene, detonation diamond soot assume the properties of non-equilibrium systems. During evaporation in the Petri dish there were found self-organized dissipative structures of micron size in the thin film of soot suspensions processed in a hydrodynamic oscillator. These structures are chain-, branched and ring-shaped cluster fractals of various dimensions, externally resembling nanotubes. The EPR method has been used to study the change in the electronic structure of soot globules after exposure to high temperatures and pressures that occur during the implosion of cavitation bubbles (cavitation activation). According to the EPR data, cavitation effects accompanying the hydrodynamic dispersion of soot suspensions lead to the deformation of the soot globules. The least significant deformations are observed in wood soot. The dynamics of dissipative carbon black structures are influenced by the structural features of black carbon globules, the nature of deformations and the presence of free and bound water. The results obtained in the course of this study can be useful in solving various nanotechnological issues.

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