

Formation of carbon submicron structures and nanostructures on the surface of cold substrates exposed to laser radiation in air

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Abstract. An experimental method is developed for preparing nanostructures in the field of high-power laser radiation ($\sim 10^7$ W cm⁻²) upon deposition of vapours of carbon-containing materials on a cold substrate surface. A specific feature of the method is irradiation of a sample in air at room temperature and pressure close to 1 atm. The dependences of the morphologic properties of nanostructures on the distance between a substrate and a sample are determined. Variations in the size of deposited nanostructures and their characteristic shape depending on the chosen material are found.

Keywords: carbon-containing materials, nanostructures, laser ablation, vapour deposition.

1. Introduction

At present nanoparticles and nanoclusters are obtained in the process of laser ablation of samples in vacuum or buffer gases [1–4]. This is mainly explained by the fact that during ablation in the atmospheric air, the carbon combustion reaction is initiated.

In this paper, we present the results of experiments on the deposition of carbon on the surface of a cold substrate by exposing carbon-containing materials of different density and different degree of graphitisation to radiation from a Nd:YAG laser.

The surfaces of substrates after exposing to laser radiation were studied with a Smena B scanning probe microscope (NT-MDT, Zelenograd). The formation of submicron structures and nanostructures was observed. The properties of these structures depend on the irradiated material and experimental conditions (laser pulse duration, exposure time, and a distance between a sample and a substrate).

2. Experimental

Carbon-containing samples (glass carbon, pyrographite, spectrally pure graphite) were irradiated by a free-running 100-W Nd:YAG laser emitting 1.5-ms pulses at a pulse repetition rate of 150 Hz. The use of this lasing regime

provided the production of a long-lived plasma over a sample surface. Figure 1 shows the deposition scheme.

The average exposure time was 30 s, the radiation power was varied from 30 to 60 W, the laser spot on a sample was 400 μ m, and the distance between the substrate and sample surface was varied from 0.5 to 2.5 mm.

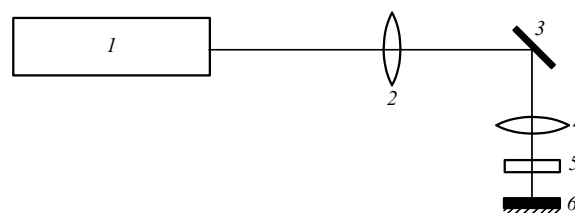


Figure 1. Scheme for the deposition of carbon vapours on a cold substrate surface: (1) Nd:YAG laser; (2, 4) focusing lenses; (3) fold mirror; (5) glass substrate; (6) carbon-containing sample.

3. Study of laser-deposited layers

After exposing to laser radiation, the surface of a substrate was studied by the methods of atomic-force microscopy by using a Smena B scanning probe microscope operating in the contact regime with the maximum scanning region of size 50 \times 50 μ m and the accuracy of measuring relief elements of ~ 10 nm in the scanning plane. We measured the surface relief and distribution of the local friction force (lateral force) because the contact regime provides higher-contrast images, which makes it possible to refine the sample relief and to analyse the surface of the evaporated layer separately from the substrate surface.

By depositing vapours produced by exposing a glass-carbon sample to the 45-W laser radiation (the exposure time was 30 s and the gap between the sample and substrate was 0.5 mm), we obtained the homogeneous distribution of the deposited substance on the substrate surface in the form of separate cones with the average height of 40 nm and the base diameter of 300 nm (Fig. 2).

We also performed experiments at the same laser power and gaps from 0.8 to 1.5 mm. No significant changes in the relief were observed. When the gap was further increased, the deposited structures were considerably rarefied, and the deposited layer completely disappeared when the gap was 2 mm.

By irradiating the surface of spectrally pure EG-2A graphite under the same conditions with the 1.5-mm gap, we observed the formation of a conglomerate of ordered structures on the cold substrate surface, which tended to

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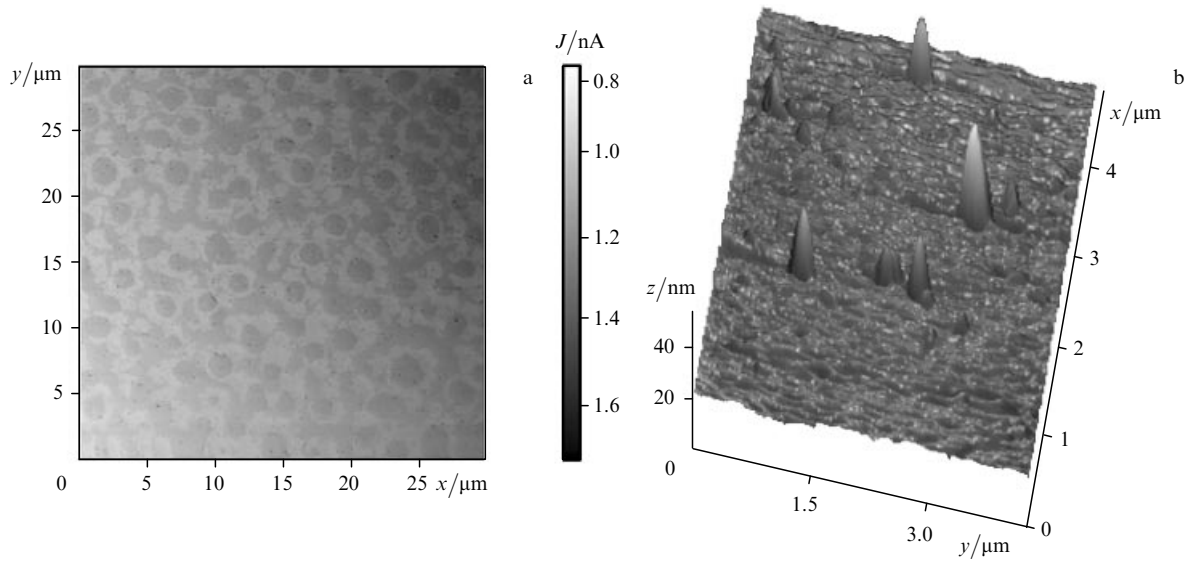


Figure 2. Structure of glass-carbon vapours deposited on a cold substrate in air: distribution of lateral forces (a) and the 3D relief of a part of the region presented in Fig. 2a (b); J is the current passed through an atomic-force microscope probe.

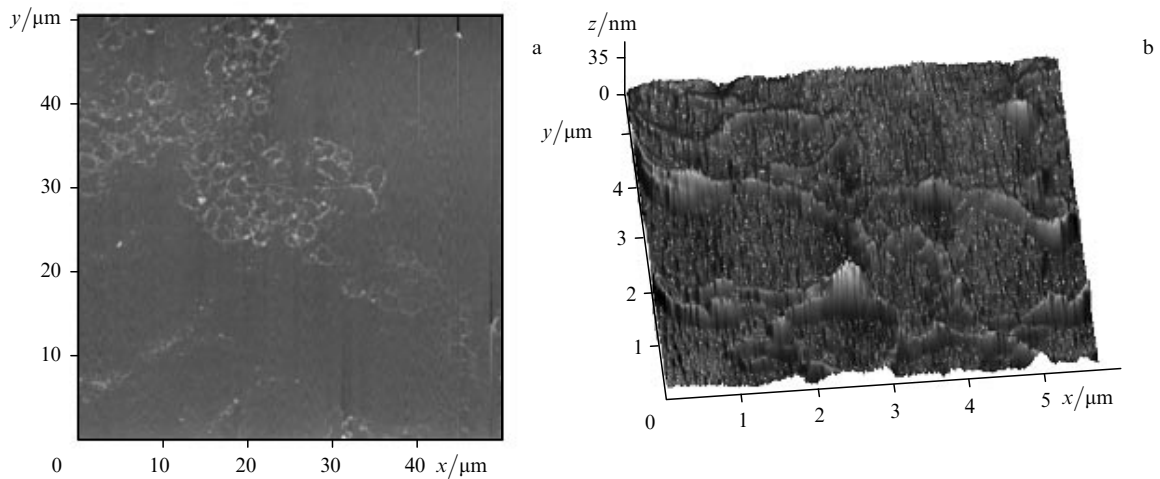


Figure 3. Structure of spectrally pure EG-2A graphite vapours deposited on a cold substrate: 2D surface relief (a) and the 3D relief of a part of the region presented in Fig. 3a (b).

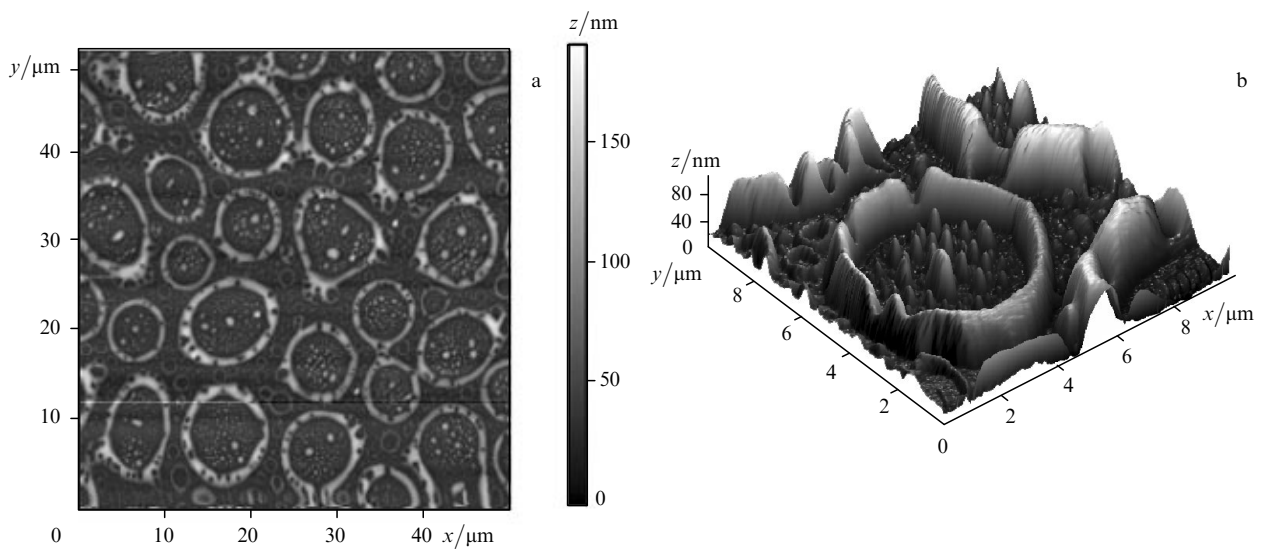


Figure 4. Structure of pyrographite vapours deposited on a cold substrate in air: 2D surface relief (a) and the 3D relief of a part of the region presented in Fig. 4a (b).

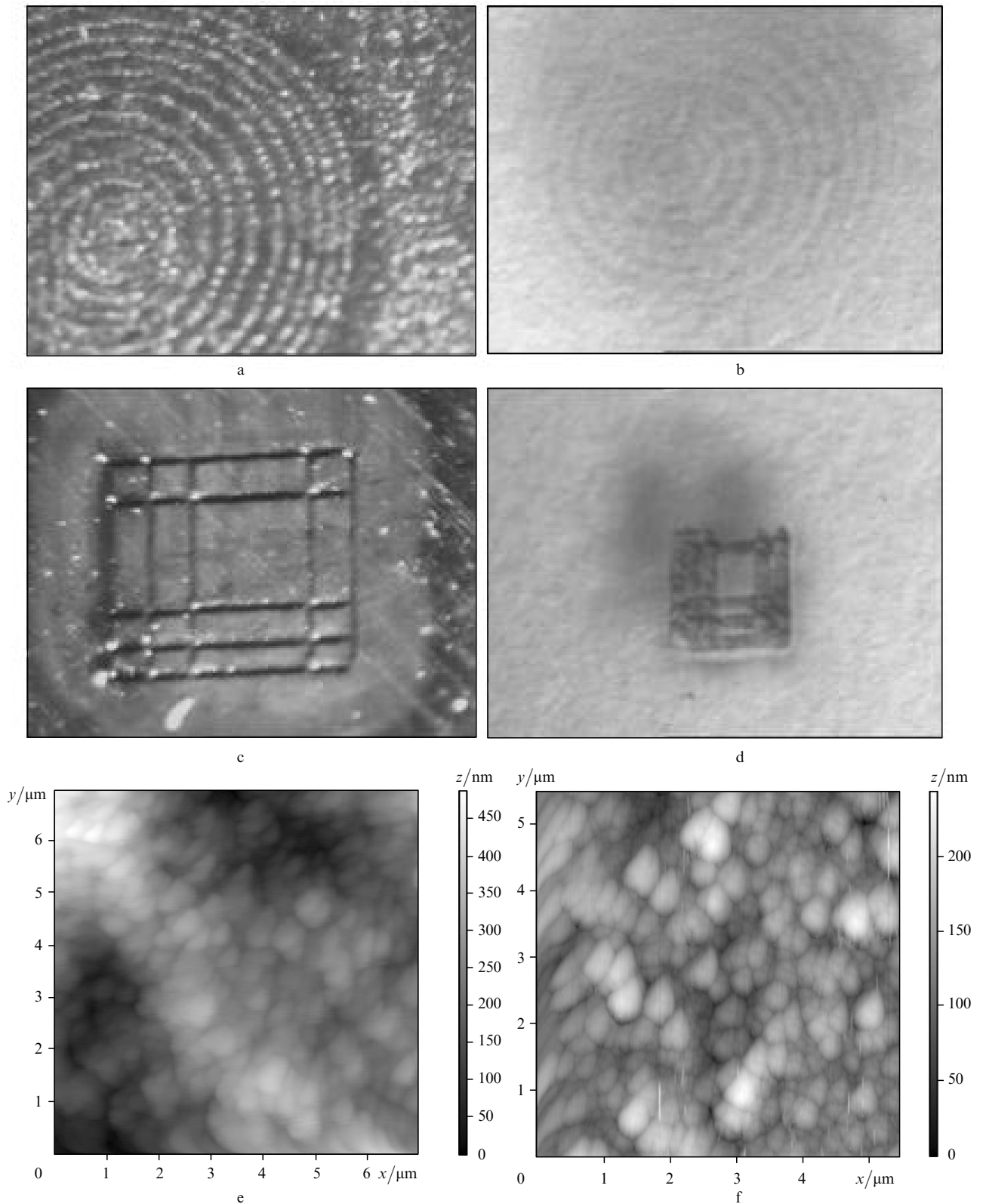


Figure 5. Images of caverns on the pyrographite surface in the form of concentric circles with the optical magnification $15\times$ (a), of the structure of pyrographite vapours deposited on a cold substrate with the optical magnification $30\times$ (b), of caverns on the pyrographite surface in the form of a lattice with the optical magnification $30\times$ (c), of the structure of pyrographite vapours deposited on a cold substrate with the optical magnification $30\times$, as well as the atomic-force microscope images of the pyrographite surface (e) and the deposited layer structure (f).

form closed elliptic structures of diameters from 3 to 5 μm and the wall height about 35 nm (Fig. 3b). As the gap between the sample and substrate was increased, first the distortion of the structure of the deposited layer was

observed, which was accompanied by a considerable decrease in the fraction of closed structures, and then a uniformly deposited layer was formed. No deposition was observed when the gap exceeded 2.5 mm.

During the deposition of carbon vapours produced by exposing the pyrographite surface to laser radiation, circular structures were formed of the substrate surface. These structures were observed most clearly when the gap between the substrate and sample was 0.8 mm (Fig. 4).

Inside circular structures of a large diameter (3–6 μm), we observed nanocones of height close to that of circular-structure walls. No additional structures were observed inside circular structures of a smaller diameter. The wall height depends on the structure diameter and varies on average from 20 to 90 μm . Such properties of the deposited layer structure suggest that it repeats the domain structure of the pyrographite surface. Thus, a flow of particles from a sample surface at small distances from it is stratified. This assumption is confirmed experimentally. For gaps exceeding 1.5 mm, no closed structures were observed on the substrate, while for gaps exceeding 2 mm, a uniformly deposited layer was observed.

To confirm this hypothesis, we performed experiments on the deposition of pyrocarbon vapours on a cold substrate lying on a sample surface. Because the heating of the substrate by free-running laser radiation caused its destruction, we used a 20-W *Q*-switched Nd:YAG laser in these experiments. The laser emitted ~ 150 -ns pulses and the laser spot diameter on the sample was ~ 50 μm .

We found that the structure of a contact-deposited layer well repeated the structure of the sample surface (Fig. 5).

4. Conclusions

Thus, we have developed the experimental method for preparing nanostructures by the deposition of carbon vapours on a cold substrate surface.

Our study of the deposition of carbon on a cold substrate by exposing carbon-containing materials to laser radiation have shown that the size and shape of nanostructures depend on the material used in experiments. To fulfil more accurate analysis, we plan to perform X-ray and Raman studies of the structural properties of deposited layers.

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