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Formation of nanostructures at the glass-carbon surface exposed to laser radiation

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***Abstract*. An experimental technique for obtaining nanostructures in the field of high-power laser radiation at the surface of carbon materials is developed. A specific feature of this technique is the formation of liquid carbon inside the region of laser action in the sample exposed to radiation in air at a pressure of ~ 1 atm. Several types of nanostructures (quasi-domains and nanopeaks) are detected in the laser cavern and beyond the range of laser action. Mechanisms of formation of such structures are proposed. The formation of quasi-domains is related to crystallisation of the melt. The nanopeak groups are formed outside the laser action region during the deposition of hot vapours of the material escaping from this region. The dependences of the variation in morphological properties of the nanostructures on the duration of laser action and the radii of typical cavern zones on the laser radiation power are obtained.**

Keywords: *glass carbon, nanostructures, laser ablation.*

1. Introduction

An investigation of the possibility of formation of nanostructures at the surface of carbon samples is currently one of the most rapidly developing trends in laser physics [1 – 4]. The development of this trend is associated with the fact that the properties of the formed nanostructures depend strongly on the parameters of laser radiation (wavelength, pulse duration and beam shape). In the laser systems developed in recent years, these parameters vary over a wide range, which gives grounds to expect that nanostructures with preset properties can be produced.

In this work, we studied glass carbon. Because of its amorphous structure, this material can be easily modified by laser radiation of intensity below 10^7 W cm^{-2}. Laser radiation intensities in this range are often used in technological laser systems at present.

Our experiments showed that the morphological properties of the produced nanostructures depend on the distance from the centre of the irradiated region. We found that the mechanisms of their formation are different. The formation of the melt at the centre of the laser cavern and the deposition of the gasous phase beyond the irradiated region were observed.

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2. Experimental

A glass carbon sample was irradiated by a 1.06-μm Nd : YAG laser emitting 1.5 – 2.5-ms pulses at a repetition rate of 150 Hz. The laser spot size on the sample was varied from 100 to 400 μm. The average radiation power was varied between 30 and 80 W, providing the radiation power density on the sample up to 10^7 W cm^{-2}. The exposure time was varied from 1 to 10 s.

3. Experimental results

The properties of samples after their exposing to laser radiation were studied with an atomic force microscope (AFM), which gave the images of the material surface. It was found in experiments that the glass carbon melting was clearly observed for the maximum irradiation power $P =$ 80 W, the exposure time no less than 3 s, and a pressure of ~ 1 atm.

Figure 1 shows the image of a typical cavern formed at the surface of glass carbon for the exposure $t < 3$ s. Our experiments showed that the process of cavern formation for $t < 3$ s has the following specific features:

(i) formation of two visually distinguishable regions: directly irradiated region (1) with a smooth relief and annular structure region (2), which scatters light strongly (Fig. 1);

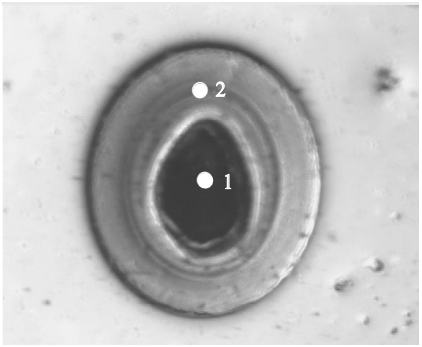

Figure 1. Image of a cavern under a magnification 28$^\times$ for power $P =$ 30 W and exposure time $t < 3$ s.

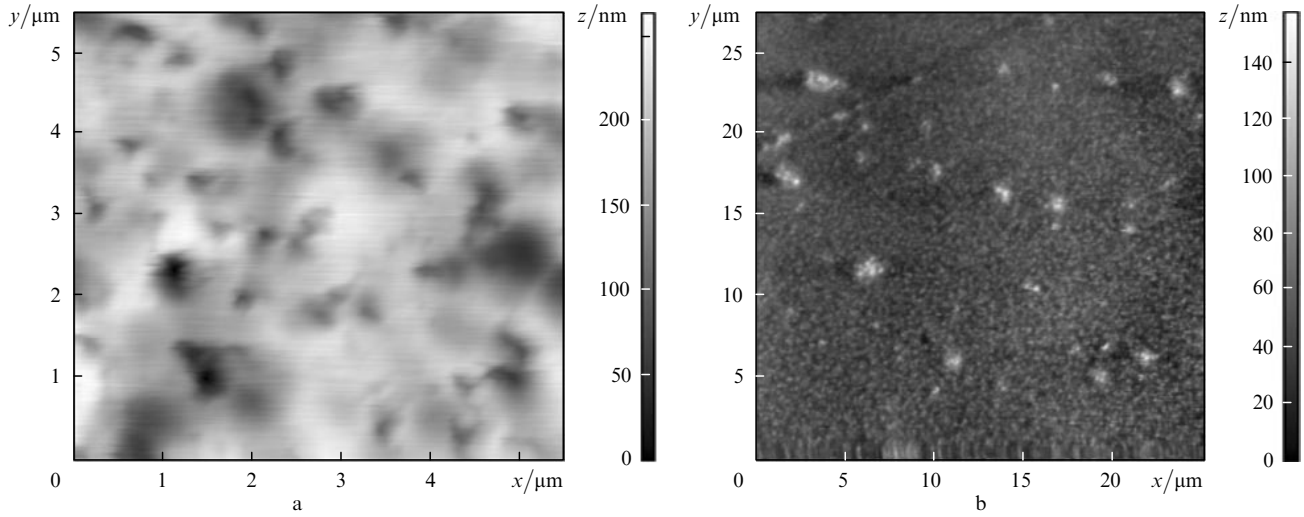

Figure 2. AFM image of the sample surface for exposure time $t < 3$ s and power $P = 80$ W: 2D-reliefs of (a) region (1) and (b) region (2).

(ii) nanostructurisation of the sample surface observed with an AFM: stalagmite structures (dips on the surface, Fig.2 a) were observed in region (1); while region (2) exhibits nanostructures in the form of peaks, which are now called nanopeaks [5] (Fig. 2b).

The size of stalagmite structures varies from 0.08 to 5 μm at the base. The average longitudinal size of these structures is 200 – 400 nm.

Nanopeaks with base diameters of 0.4 – 0.5 μm and heights of 60 – 300 nm represent isolated structures and are located in the vicinity of the central ring. Denser formations with a lower height and a broader base are observed towards the outer edge.

An increase in the exposure time ($t > 3$ s) led to an increase in the number of visually distinguishable regions in which qualitative variation of the surface relief could be traced during scanning. This variation was manifested most clearly in a change in the diameter of the observed region as a whole, the depth of the central zone of the cavern, the height of the relief in the transition regions, and in the formation of a large number of fractures in the central zone (Fig. 3).

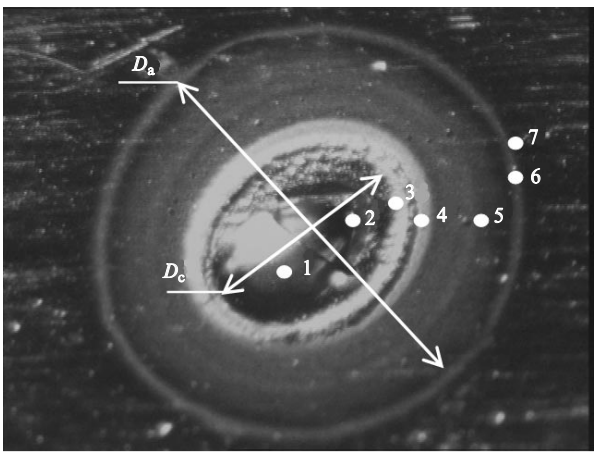

Figure 3. Image of a cavern on the glass carbon surface under a magnification $28^{\times}$ for power $P = 76$ W and exposure time $t = 5$ s; D_a and D_c are the diameters of the entire region observed and the cavern, respectively.

Remelted carbon is located in region (1), where the difference in the relief heights is quite large (Fig. 4a). Nearly regular quasi-domain structures are observed in regions (2) and (3) (Figs 4b and 4c). Because of a high recurrence of the structures, their image resembles nanograins on the surface of materials treated at high pressures and at high temperatures [1, 3]. The difference in the shapes of quasi-domain walls suggests that such domains were formed in different processes. The formation of distinct regular polygons (in our case, pentagons and hexagons) in region (3) leads to the conclusion that, in conformilty with [3, 6], a thin layer of homogeneous liquid crystallises on the amorphous surface. The destruction of regular quasi-domain walls in region (2) is probably caused by the temperature effect [3].

Annular structures are formed at the cavern boundary [region (4)]. The surface between the rings is strongly inhomogeneous, and a large number of 'crimps' and nanoroughnesses are observed (Fig. 4d).

Nanopeaks were observed in the regions (5), (6) and (7), and a transition region could be singled out on the sample surface. This region is distinguished by the presence of the initial relief of the sample under new formations (Fig. 5). The region has distinct boundaries and its size depends on the power and duration of the laser action. The formation of the transition region is probably associated with deposition of hot vapour of the material escaping from the irradiated region. It can be stated that the destruction of the surface in the solid phase under the action of the thermal stresses in this case is not a dominating mechanism since the initial surface relief is preserved.

In the series of experiments described here, radiation from the Nd : YAG laser was incident at an angle to the normal to the sample surface, which made it possible to trace the efflux of the liquid carbon phase in the direction of the melt channel formed under the action of the recoil vapour pressure [7] (see Fig. 3). However, nanopeaks observed in the AFM are formed on the entire surface of annular regions (5), (6), (7) at nearly the same rate. Hence it can be stated that crystallisation of the melt does not affect the formation of these structures.

Our experiments have shown that variations in the morphological properties depend on the exposure time. We have failed to obtain the quantitative dependence of

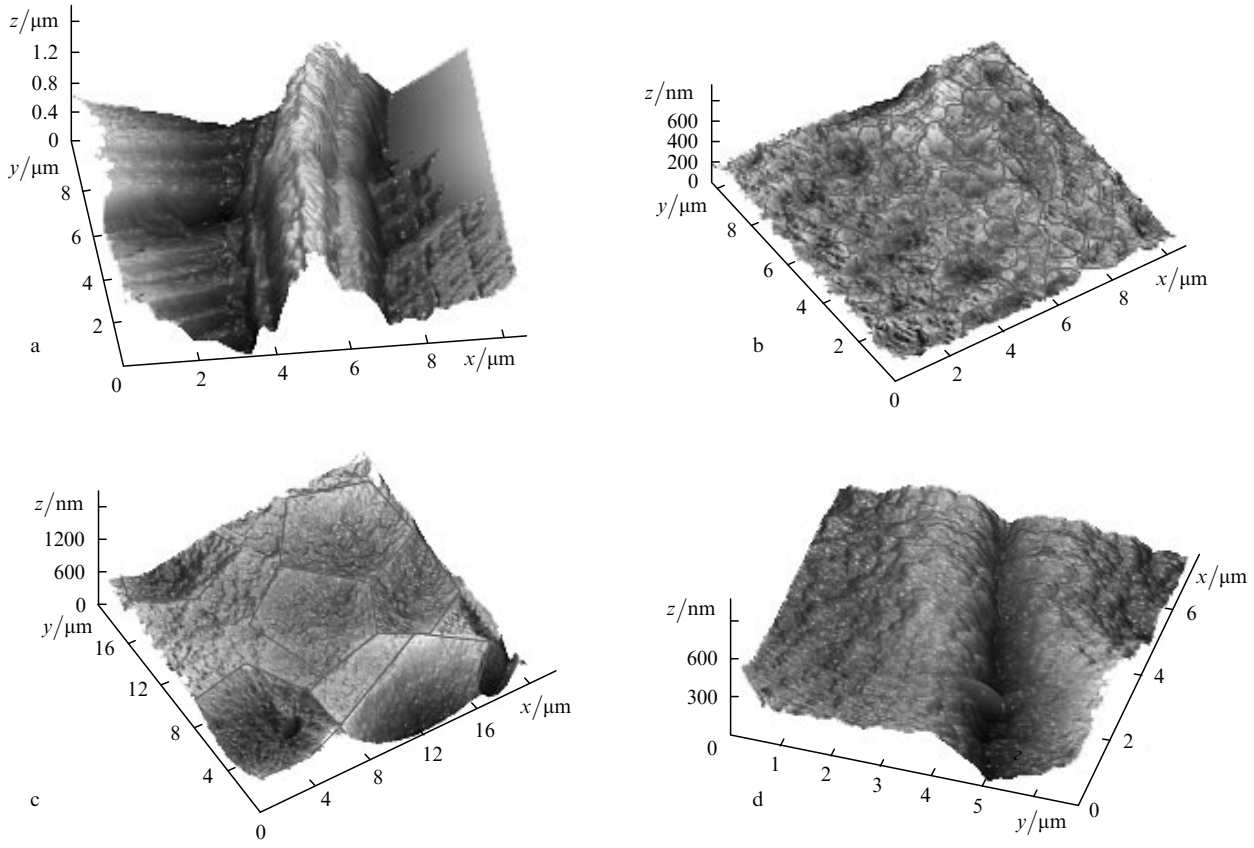

Figure 4. AFM image of the sample surface in various regions: (a) central region; (b) region at a distance of ~ 50 μm from the centre; (c) at the inner and (d) outer boundaries of the cave.

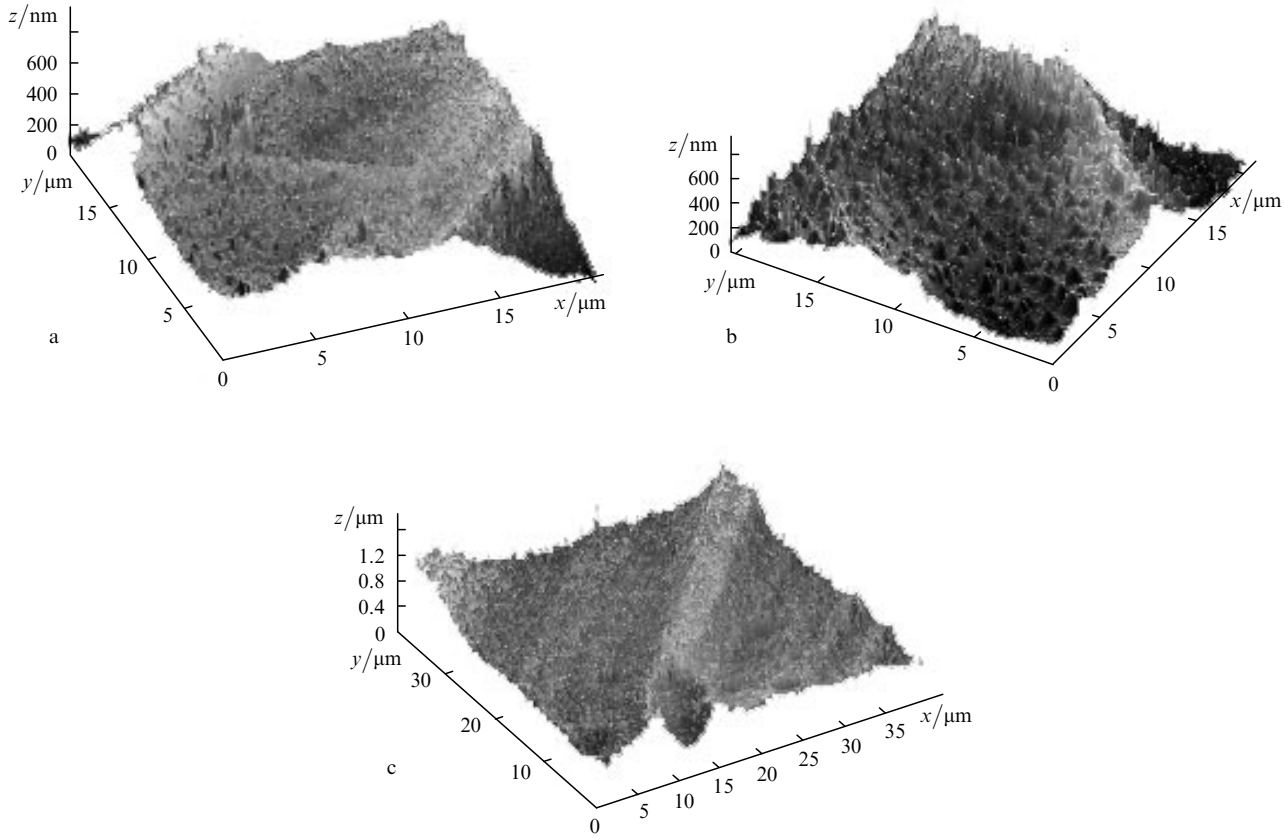

Figure 5. Nanopeaks in (a) region (5) (deposition region); (b) region (6) (formation of the outer rim); and (c) region (7) (deposition on the initial surface).

these properties on the laser radiation power because the diameters of typical regions increased with laser power (see Fig. 3). Figure 6 shows the dependences of the cavern diameters and the modified surface region on the radiation power. Thus, we have established experimentally that variations in the laser power at a fixed exposure time do not affect the geometrical parameters of nanostructures, but determine their spatial distribution.

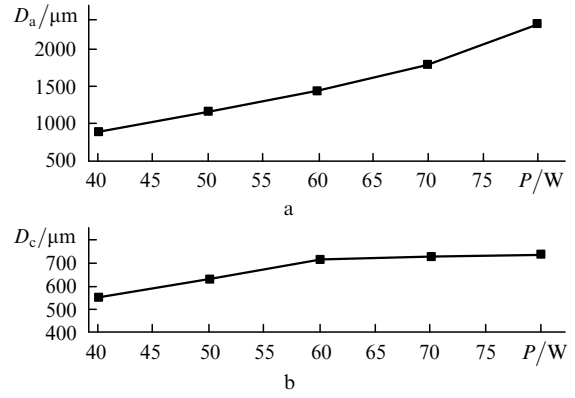

Figure 6. Dependences of the diameters of (a) the entire modified area and (b) cavern on the laser radiation power.

To measure the geometrical dimensions of the nanopeaks formed outside the irradiated region, clearly distinguishable isolated nanostructures were singled out on the obtained relief of the surface. The dependences presented in Fig. 7 show that the increase in the mean diameter of the nanopeaks with the exposure time terminates at $t = 5$ s. Such a behaviour agrees with the above theory of formation of nanostructures during deposition of hot vapours.

Variations in the heights of nanopeaks terminates with time, and the average height of the formed structures remains unchanged at $t \geqslant 6$ s (see Fig. 7b). This is most likely explained by the fact that the surface is uniformly covered by the deposited carbon atoms during long exposures.

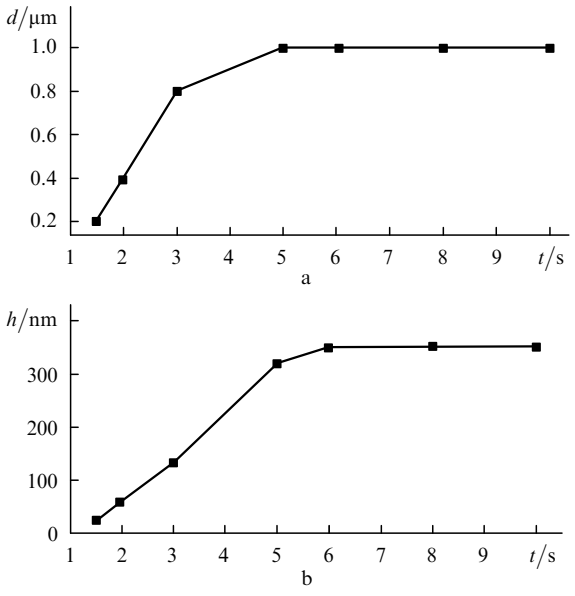

Figure 7. Dependences of (a) the mean FWHM diameter d of nanopeaks and (b) their mean height h on the exposure time.

4. Conclusions

We have developed the experimental technique for obtaining nanostructures at the glass carbon surface in the high-power laser radiation field. A specific feature of this technique is the formation of liquid carbon inside the region irradiated in air at a pressure of ~ 1 atm.

Nanostructures of different types have been observed inside a laser-produced cavern and outside the irradiated region. The typical form of the structures detected near the centre of irradiation suggests that the liquid phase is formed during laser action.

Quasi-domains are probably formed during crystallisation of the melt [4], while the formation of groups of nanopeaks near annular structures is caused by microscopic melting and mass transfer processes [5, 8]. We have found that groups of nanopeaks observed outside the irradiated region are formed due to deposition of hot carbon vapour on the cold surface.

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