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Influence of physical effects on the structure of soot particles of hydrocarbon flames

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Abstract. The structure of the diffusion flame of TC-1 kerosene has been studied. A film of pyrolytic soot was obtained using the sampling method, and the structural changes of this film under thermal exposure were studied. A new carbon structure – glass carbon was obtained as a result of laser irradiation of highly dispersed carbon black. The mechanism of bubble formation in the glassy carbon structure has been established, and the latent heat of carbon "melting" equal to 2110 J/kg has been determined.

Keywords: flame, soot, carbon, pyrolysis, IR-spectroscopy

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Влияние физических воздействий на структуру сажевых частиц углеводородных пламен

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Аннотация. Проведено исследование структуры диффузионного пламени керосина марки ТС-1. Посредством метода пробоотбора получена пленка пиролитической сажи, изучены структурные изменения данной пленки при термическом воздействии. В результате лазерного облучения высокодисперсной сажи получена новая углеродная структура – стеклоуглерод. Установлен механизм формирования пузырьков в структуре стеклоуглерода, определена скрытая теплота «плавления» углерода, равная 2110 Дж/кг.

Ключевые слова: пламя, сажа, углерод, пиролиз, ИК-спектроскопия

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Introduction

Soot particles are formed during the combustion of hydrocarbon fuels. Despite numerous studies of the morphology and structure of soot particles, the mechanism of their formation is still not fully understood [1, 2, 3]. According to one hypothesis, soot particles are formed as a result of the hydrogenation of hydrocarbon molecules, followed by their combination into a single particle. Primary soot particles are described as amorphous carbon. In contrast, another hypothesis suggests the formation of more complex molecular structures, in particular polycyclic aromatic hydrocarbons, which serve as nuclei for the formation of carbon black particles.

Materials and Methods

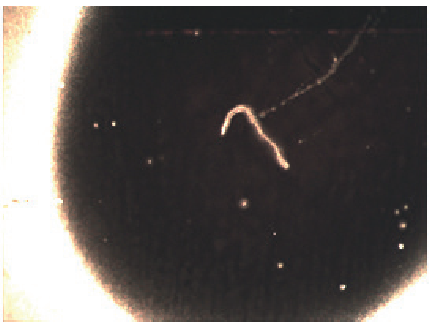
Light fractions of oil (TC–1 fuel) burn to form a diffusive luminous flame. The shape of the flame is determined by the design of the burner device. In the case of a cylindrical burner, the formation of a conical-shaped diffusion flame is observed. At the base of the flare, the mixture formation is such that the hydrocarbon fuel burns almost in a stoichiometric ratio. The position of the combustion front of the flame is close to the side surface of the cone. With increasing altitude, due to the lack of injected oxygen, the completeness of combustion decreases, highly dispersed soot particles are formed from fuel molecules, the glow of which turns the torch yellow-orange. A plume of sooty particles forms at the top of the torch. There is a zone near the top of the flare cone, where the density of the heat flow from the combustion front is high. Volumetric pyrolysis of a combustible substance without ignition is possible in this zone. Pyrolysis products can condense on the surface of soot particles in the plume area at the top of the flare. Pyrolysis products also occur at the boundary adjacent to the combustion zone. They can undergo further oxidation, when pass through the combustion zone.

Results and Discussion

The introduction of the sampler into the volumetric pyrolysis zone creates conditions for pyrolysis products to settle on the surface of the sampler. In the central region of the flame, in the zone of active pyrolysis, carbon was deposited on the surface by a film-island mechanism, on the periphery of the flame – in the form of highly dispersed particles.

Examination of the film surface using an optical microscope shows the absence of pronounced irregularities. However, when the sampler is heated, the film begins to break apart and peel off from the glass (Fig. 1). The lines of discontinuity are characterized by the absence of pronounced symmetry. Eventually, a pile of detached film fragments forms in place of the film.

a)



b)

Fig. 1. Pyrolytic carbon film before thermal exposure (*a*); after thermal exposure (*b*)

The use of an atomic force microscope SolverNext made it possible to obtain a micro relief of the film surface (Fig. 2). The film consists of a set of soot particles in contact with each other, having a predominantly rounded shape. The particle sizes range from 200 to 300 nm, but there are also larger formations reaching 1300 nm in diameter. The surface of the particles, as a rule, is not characterized by a spherical shape, but consists of layers.

a) *b*)

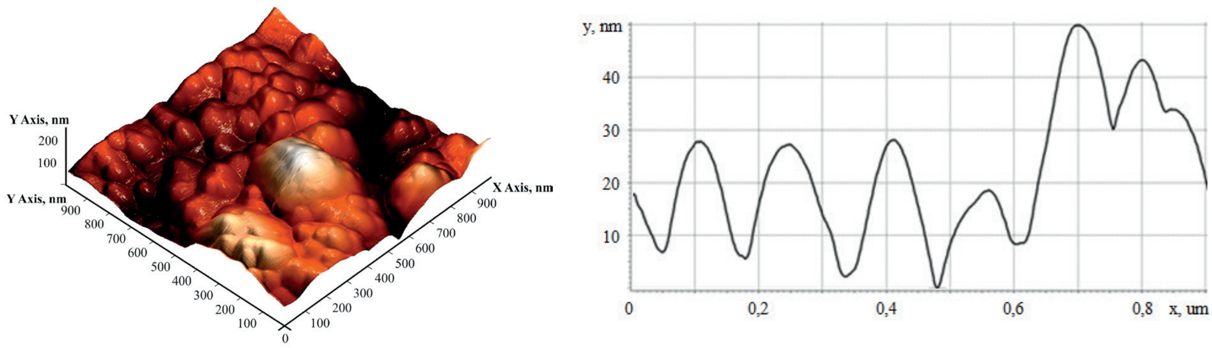


Fig. 2. Surface of pyrolytic film on AFM: 3D—surface (*a*); surface profile at x = 800 nm (*b*)

The space between the particles is filled with binder. However, break lines representing structural inhomogeneities (defects) are detected. Thermal influence on the sampler leads to the fact that the film destruction starts in the areas corresponding to these defects.

Spectral analysis of soot samples obtained using the FSM—1201 spectrometer showed continuous absorption of radiation in the entire studied wavelength range [4], from 4000 to 500 cm^{-1}. Similar results were obtained for industrial carbon black samples, in particular, PM-50 and LM-15 grades. During the production process, industrial carbon black undergoes a stabilization stage, during which active centers such as CH-groups and oxygen-containing compounds are removed. Further it provides its long-term storage without significant changes in characteristics.

In the work by Prikhodko N.G., Mansurova Z.A. [2] describes the process of nucleation of carbon nanostructures in the form of C_{60} and C_{70} fullerenes in a hydrocarbon flame at low pressures. At the initial moment, amorphous carbon is formed, from which graphene plates are then formed. Graphene plates, folding, form fullerene molecules. The author emphasizes that the ratio between C_{60} and C_{70} molecules can be controlled. There is no information about the properties of amorphous carbon in the work.

Murga M.S. [3] considers the structure of amorphous carbon as a medium with a short-range order in the arrangement of atoms. It is hypothesized that amorphous carbon in the intergraphine space may exhibit properties characteristic of the liquid phase.

Spherical particles of highly dispersed carbon black consist of a set of individual crystallites [4, 5]. Within each crystallite, the arrangement of carbon atoms is ordered. However, within a spherical particle, the relative arrangement of crystallites is chaotic.

When exposed to laser radiation, the structure of highly dispersed carbon black changes. A semiconductor OCG at a wavelength of 450 nm with a beam power of 4.5 W was used as a laser. When focusing with the output lens, you can get a spot with a diameter of 0.33 mm. Highly dispersed soot within the beam turns into a glassy mass. Some of the carbon is oxidized in the air and forms a gas that inflates the glassy substance into a bubble. Nearby, the bubbles form thin walls between each other, which are easily visualized when viewed with an optical microscope (Fig. 3, *a*). The resulting carbon dioxide diffuses through the pores in the film and leaves the bubble cavity.

Further study of the structure of glass carbon and highly dispersed carbon black was carried out using a scanning electron microscope JQSCAN W—32. Microphotographs of the sampler sections were obtained (Fig. 3, *b*, *c*), the elemental composition of dispersed particles was studied. The film is characterized by a porous structure with pore sizes ranging from 250 to 1250 nm. At a magnification of 50000x, the internal structure of the film itself can be seen. It is a spatial mesh consisting of nanoparticles of 30—60 nm in size and interconnected by thin junctions. Highly dispersed carbon black consists of particles that vary in size over a wide range from 50 to 230 nm. The particles are in contact with each other but do not merge.

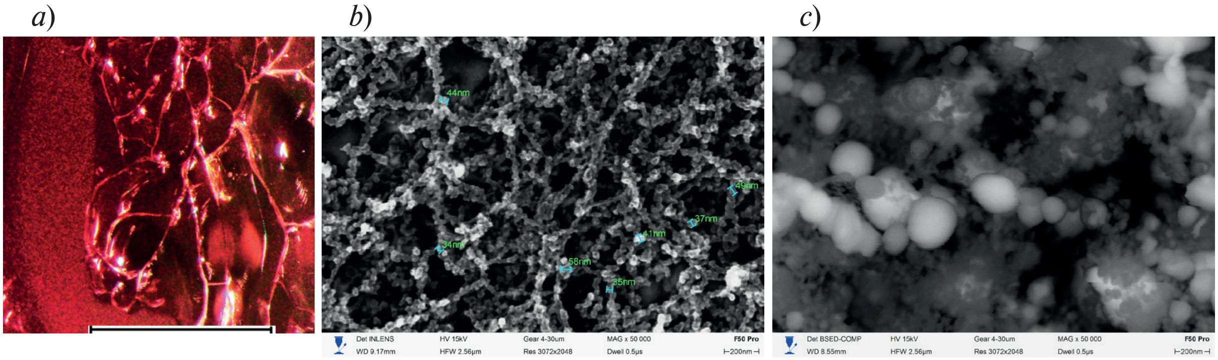


Fig. 3. The surface of the sampler section: the structure of a glass–carbon bubbles – optical microscope (reference line 1 mm) (*a*); scanning electron microscope (50000x) (*b*); the structure of deposited highly dispersed carbon black – scanning electron microscope (50000x) (*c*)

X-ray microanalysis allowed to determine the elemental composition of the studied samples. In particular, the mass fraction of carbon in glass carbon amounted to 23.6%, in highly dispersed carbon black – 25.16%. It was found that under the influence of laser radiation 1.56% of carbon black is burned out with the formation of carbon dioxide, which, expanding, forms a bubble in the glass-carbon matrix.

Thus, the energy of laser radiation is spent on the process of carbon black burnout, as well as on the phase transition of carbon black crystallites into the liquid phase – glass-carbon. The latent heat of carbon "melting" turned out to be equal to 2110 J/kg. The obtained results require further investigation. Literature sources [6] contain data on the heats of phase transitions of graphite and diamond, the values of which are 1000 and 5000 J/kg, respectively.

Glass carbon is produced on an industrial scale by pyrolysis sintering from phenolic resins in vacuum and is subsequently used as a chemically inert structural material capable of withstanding high temperatures [6, 7].

Conclusion

1. A film of pyrolytic soot was obtained by the method of passing a glass plate through a flame, and the structural changes of this film under thermal influence were studied.

2. It has been established that as a result of the effect of optical radiation on highly dispersed carbon black a new carbon structure – vitreous carbon – is formed.

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