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Study of the generation of a weakly ionized medium for spraying of biocompatible coatings by two-stage pulsed plasma set

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Abstract. The paper is devoted to the experimental study of the generation of the weakly ionized high velocity heterophasic flows by gas detonation in acetylene-oxygen mixtures. Such flow can contain fine hydroxyapapite (HOA) particles with an average diameter of $d_p \leq 100$ µm for spraying of biocompatible coatings. The characteristic velocity (≈2.3…2.5 km/s) and pressure (≈3.1…3.8 MPa) of the detonation waves were defined and the attenuation dependences of those parameters were stated. The structure of the detonation flows was visualized and the chemical composition was estimated. The samples of the HOA coatings (with a thickness of 80…100 µm) were prepared on the carbon nanocomposites and their properties were characterized by SEM, EDX and XRD. The possibility of the using of such flow as a working fluid for electromagnetic acceleration of HOA was offered by the two-stage pulsed plasma set.

1. Introduction

Nowadays the using of the two-stage pulsed plasma sets [1] for coating spraying systems is under consideration. A weakly ionized (with a degree of ionization of $\approx 10^{-4} \ldots 10^{-3}$) detonation wave (DW) is formed by gaseous detonation [2] within a cylindrical barrel at the first stage. Additional acceleration of the detonation products takes place due to discharging of capacities into this wave during the second stage within a coaxial electrode unit. Owing to Joule heating generation of a conductive plasma channel with a temperature of 20…30 kK occurs. The channel expends [3] with a supersonic velocity and generate of a shock wave. The using of the preliminary detonation allows decreasing energy consumption for this acceleration within the coaxial electrode unit. The particles which were injected before the wave front are accelerated up to velocities of 1.4…2.0 km/s and transferred to a substrate where they form a coating.

Note that the complications of the flows generation processes with non organic high-molecular particles can limit the efficiency. As an example of this method spraying biocompatible coatings [4] from hydroxyapatite (HOA) $Ca_{10}(PO_4)_6(OH)_2$ was studied in this paper. As substrates the carbon nanocomposites [5] were used. The detonation with the regime of weak wave generation can decrease the dynamic loads on to the substrate and avoid its crash.

2. Experimental set

Based on commercial CCDS 2000 equipment [2] the experimental set working with C_2H_2 and O_2 mixtures as the fuel was designed (see figure 1). The facility "Beam-M" [6] was used for the flow characterization. During the experiments the fuel composition was stoichiometric. The generation of the detonation flows was occurred within a cylindrical barrel (1) with the diameter of d=1.6 cm by a spark (2). The set was equipped by a gas feed system (3). The moving of non-dusty flows and heterophasic (with an average diameter of HOA particles of $d_p \approx 70$ nm and 100 μm) flows was studied. An injection of HOA was realized into the direction normal to the barrel axe by a pneumatic feeder (4). A volume flow rate of HOA was about of 0.3…1.0 mm^3 per a shot. Piezometric sensors were used for control of the flow dynamics. The one (5) was built in the barrel at the injection zone and the other (6) was embedded into a substrate (7). The distance δ between the muzzle and the substrate was varied from 2.5 to 23 cm. The distance l between the injection zone and the muzzle was l=40 cm. The data recording from the sensors was realized by a storage oscilloscope (8) with the using of a syncronizing signal from the spark. The flow structure was visualized by a laser Schlieren photography. A Nd:YAG laser (10) with the wavelength of λ=532 nm was applied as a source of the radiation that was fixed by a high-speed camera (11). The polarizing filter (12) was used for the regulation of an intensity of the laser radiation. The system consisting of an interference filter (13) with λ_{max}=532 nm and $\Delta\lambda$=10 nm and a diaphragm (14) was used for cutoff of the flow (9) radiation and visualization of flow disturbances. Producing of a collimated laser beam was done by lenses (15) and (16). Delay of a picture capturing by the camera was set relativity the sensor (5) by BNC 575 synchronizing unit (17). A part of the flow radiation was transmitted by prism (18) to Solar SC125 spectrometer (19) for its characterization.

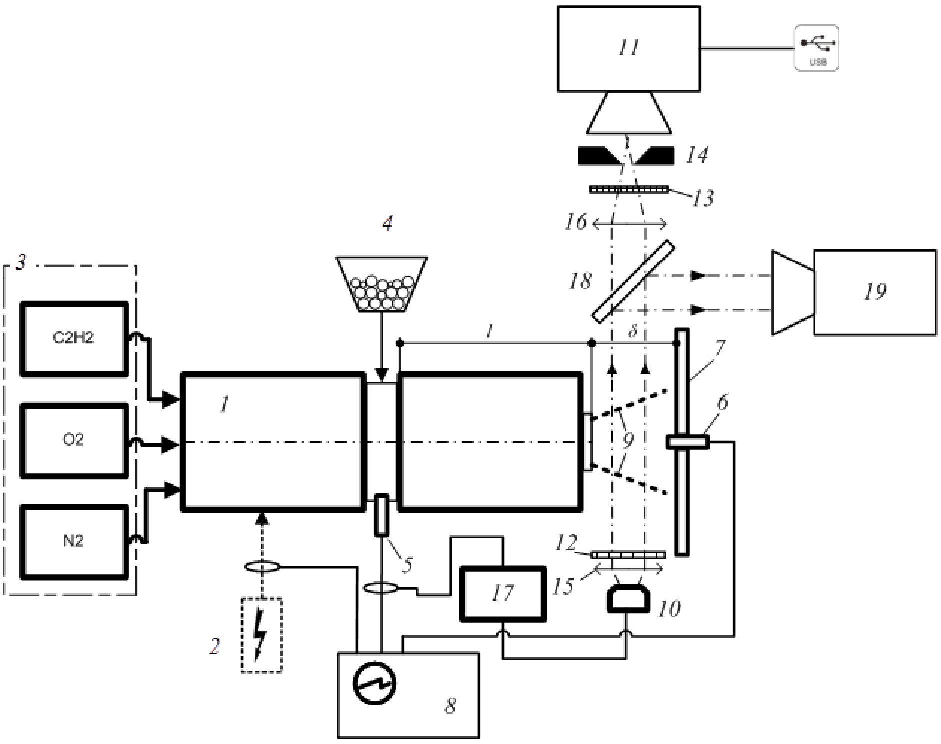
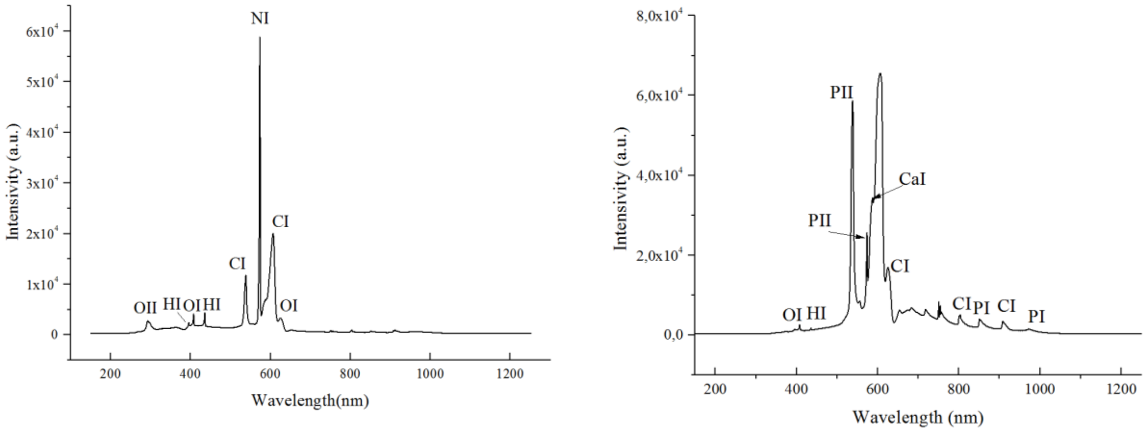


Figure 1. Experimental set: 1 – barrel; 2 – spark; 3 - gas feed system; 4 – pneumatic HOA feeder; 5 and 6 – piezometric sensors; 7 – substrate; 8 – oscilloscope; 9 – detonation flow; 10 - Nd:YAG laser; 11 – high-speed camera; 12 - polarizing filter; 13 - interference filter; 14 – diaphragm; 15 and 16 – lenses; 17 - BNC 575 synchronizing unit; 18 – prism; 19 - Solar SC125 spectrometer.

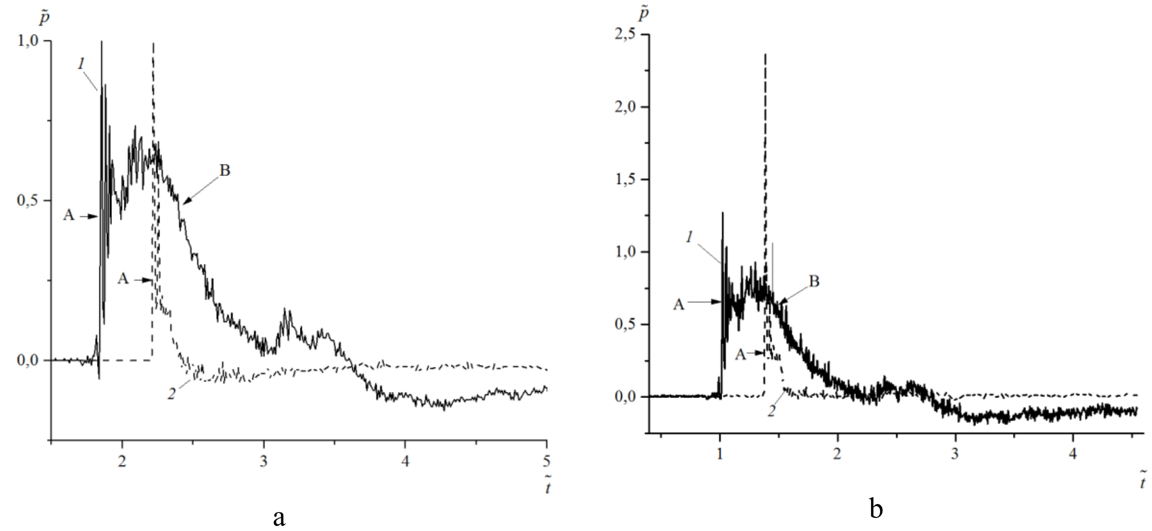
3. Results and discussion

Spectroscopy (in the wavelength range of 150…1250 nm) of the non-dusty flows at *δ/l*=3.13 showed (see figure 2) the presence of the intensive lines of atoms and first ions of C, O and atomic H. In a case of HOA injection with $d_p \approx 70$ nm the specter contained also further the lines of atoms and ions of P and Ca. Furthermore, increasing of the intensity of atomic O was detected. Such circumstances evidence about partial pyrolysis of HOA.



a b

Figure 2. Spectra of non-dusty flow (a) and heterophasic flow (b) with $d_p \approx 70$ nm.



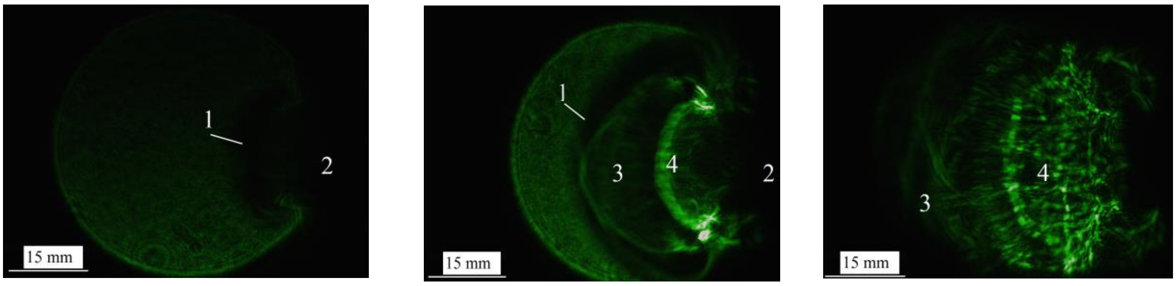
a b

Figure 3. Dynamics of pressure for non-dusty flow (a) and heterophasic flow with $d_p \approx 70$ nm (b): 1 - $\tilde{p}\left(\tilde{t}\right)$ at the injection zone; 2 - $\tilde{p}\left(\tilde{t}\right)$ at the distance of *δ/l* from the muzzle.

Data from the piezometric sensor (5) demonstrated (see figure 3) registration of the strong DW (A) which propagates and compresses the non-disturbed fuel-oxygen mixture. Thereby the initiation of intensive combustion occurs which was indicated as pressure decreasing (B). In figure 4 the dependences of dimensionless pressure $\tilde{p}\left(\tilde{t}\right)$ are presented. Here $\tilde{p} = p / p_0$ and $\tilde{t} = t / t_0$ is dimensionless time, where $p_0 = 3.1$ MPa and $t_0 = 440$ μs. It was shown that the amplitude $\tilde{p}\left(\tilde{t}\right)$ of the heterophasic DW within the injection zone is ≈1.1…1.25 times higher than of non-dusty flows because of the turbulization.

The sensor (6) demonstrated that DW transforms into a strong shock wave (SW) at the muzzle. The SW attenuates with δ/l increasing and becomes weak at $\delta/l>7$. The characteristic values of heterophasic flow velocities ($D_0\approx2.3\ldots2.5$ km/s) at the muzzle were estimated. Note that for the flows with HOA the pressure pulse is 1.5…3.5 times higher owning to the action of HOA particles on to the substrate.

Schlieren photography demonstrated the features of structure and dynamics of the non-dusty flows and the heterophasic flows with HOA particles ($d_p\approx70$ nm). It was shown the absence of influence of such particles on the DW structure at the provided volume flow rates. In figure 4 the photos of the detonation in flowing (from left to right) are presented at the varied delay time t_d. The diffraction of the wave front (1) at the muzzle (2), the shock-compressed mixture (3) of the fuel and oxygen and high brightness region (4) of combustion were visualized.



a b c

Figure 4. Dynamics of the flow structure at varied t_d: 1 – wave front; 2 – muzzle; 3 – shock-compressed layer and 4 – combustion region; a - t_d=170 µs; b – 210 µs; and c – 220 µs

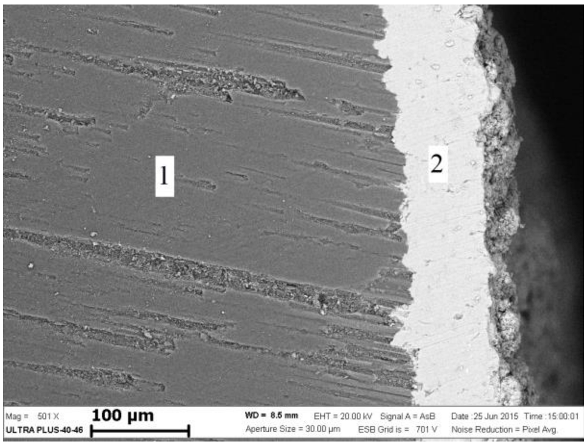


Figure 5. SEM image of the sample: 1 – carbon substrate; 2 – HOA film

With the using of the set the samples of the coatings (with thickness of 80…100 µm) on the carbon substrates were prepared. The coating morphology (see figure 5) was studied by SEM with Zeiss Ultra plus 55. This has a close packed structure without visible defects. EDX and XRD (DRON 3M diffractometer) showed that the coatings are polycrystalline with Ca and P concentration ratio of ≈1.67 that corresponds to raw HOA.

4. Conclusion

The interaction of the detonation flows with fine HOA particles was studied. It was shown that the produced flow is high velocity weak ionized medium. This medium is available for electromagnetic acceleration within the coaxial electrode unit of the two-stage pulsed plasma set for spraying of biocompatible coatings.

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Acknowledgments

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