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"PHYSICS AND DIAGNOSTICS OF LABORATORY AND
ASTROPHYSICAL PLASMAS" (PDP-11)**

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Edited by A.N. Chumakov, M.M. Kuraica and M.S. Usachonak

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ИНСТИТУТ ФИЗИКИ ИМЕНИ Б.И.СТЕПАНОВА**

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"ФИЗИКА И ДИАГНОСТИКА ЛАБОРАТОРНОЙ И
АСТРОФИЗИЧЕСКОЙ ПЛАЗМЫ" (ФДП-11)**

15–19 декабря 2016 г., Минск, Беларусь

Под редакцией А.Н. Чумакова, М.М. Кураицы и М.С. Усачёнка

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FORMATION OF MICROSTRUCTURES AND OXIDES ON STEEL SURFACE BY LASER IRRADIATION IN AIR AND LIQUIDS

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Abstract. Features of submicron structures and oxides formation on steel in air and liquids under laser irradiation are investigated experimentally.

Interaction of laser radiation with steel surface near the ablation threshold can lead to improve mechanical, optical and adhesion properties of modified material, due to surface nano and micro structuring, as well as formation of metal oxides /1–5/.

The most widespread theories of surface nano- and microstructuring under the action of laser radiation are often associated with the generation of surface electromagnetic waves, defect-deformational structuring of solids and multiple recrystallization of the molten layer /6–8/.

Should be noted that the dynamics of laser ablation plasma significantly affects on the surface structuring due to the formation of condensed phase in the expanding ablation plasma and deposition of small particles on the target surface /9/. Moreover, several studies devoted to the laser surface modification in a liquids observed that the liquid surrounding medium plays an important role in the generation of surface structures and reducing their size, as compared with the air and other gases /4-5/. This phenomenon may be associated with the features of the dynamics of laser plasma in the liquids.

The purpose of this paper is to identify regularities of producing submicron structures and oxides on the steel surfaces by laser irradiation in air and liquids with the fluences are exceed the ablation threshold.

First, the stainless steel samples were irradiated by laser pulses ($\tau \sim 20$ ns, $f \sim 10$ Hz, $\lambda = 532$ nm) at power density of $0.5 \cdot 10^9 \div 0.7 \cdot 10^9$ W/cm², which is close to the ablation threshold in the air. Surface morphology of the irradiated samples in the different areas of laser spot was analyzed by scanning electron microscopy (SEM). Investigation of the laser-induced morphological changes in the steel surface has shown their dependence on the laser beam characteristics, the number of laser pulses, power and energy density of laser radiation, etc. Significant differences were found between laser-induced structures in the center of the laser spot, at its edges, and in the nearest surrounding of the spot.

It was found that the surface structures in the center of the spot vary on the number of laser pulses. Irregular microstructures were prevailed after the series of 10 laser pulses (Fig. 1, A1) and pores 200 nm to 2 μm in diameter were observed after the 100 laser pulses (Fig. 1, A2). This phenomenon may be associated with multiple recrystallization of molten layer in the central area of the laser spot. Besides, an increase of surface microhardness (Table 1) with increasing number of laser pulses up to 2000 was found at the central area of the spot, where the power density of laser radiation reaches its maximum, and it is very useful for the development of strengthening technologies.

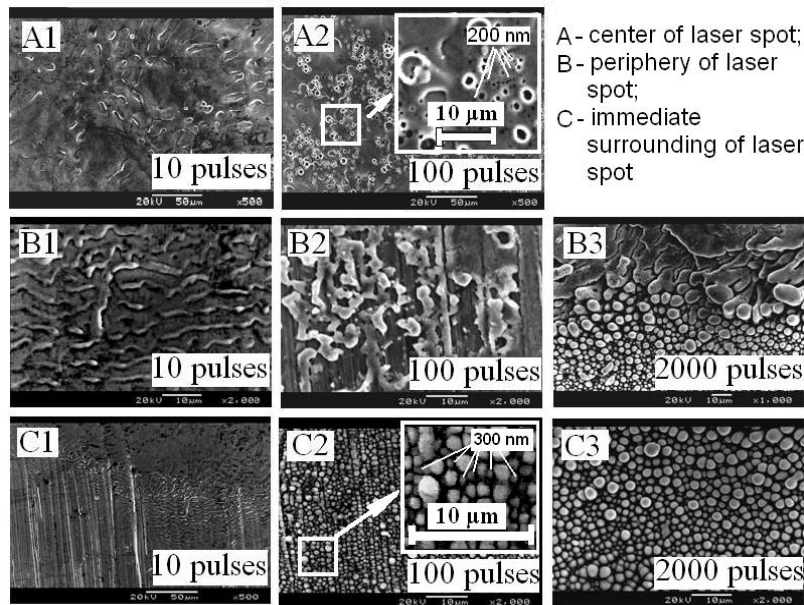


Fig. 1. SEM images of nano- and microstructures on modified steel surface

Table 1. Microhardness of initial and modified steel samples

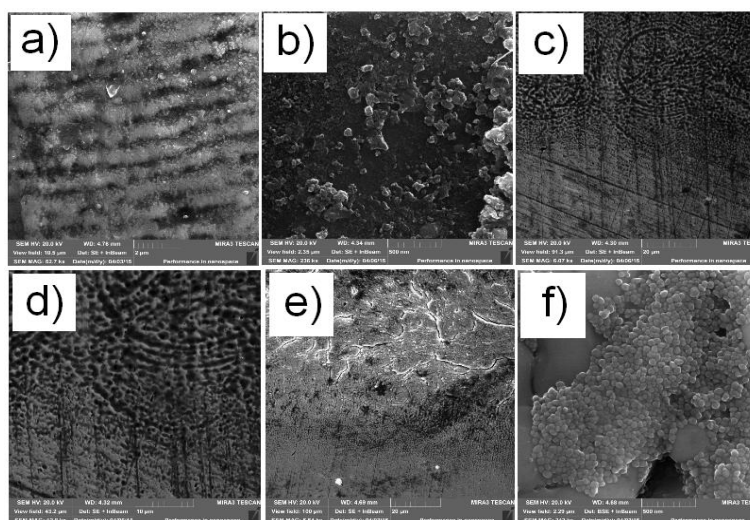
Stainless steel (MnNiCrMo-steel and MnNiCrMo-steel with Ti)	Microhardness HV, $\pm 0,2$ GPa	
	Load 10 g	Load 20 g
Initial sample	6,98	6,27
Modified sample (center of spot, 2000 laser pulses)	9,71	6,66

Wave-like structures, irregular microstructures, and a grain structure (after series of 10, 100, and 2000 pulses respectively) have been revealed at the periphery of the laser spot (Fig. 1, B). The formation of the regular grain structures at the periphery of the laser spot and in its immediate surrounding after 100–2000 laser pulses (Fig. 1, C2, C3) is of great interest for improving

optical properties of the surface, its adhesion characteristics and a biocompatibility of the material. The regular grain structure widely ranges in size from 300 nm to 10 microns. A fraction of particles with dimensions above 1 micron significantly increases with the number of laser pulses varying from 100 to 2000. These results show an increasing the number of small particles deposited due to condensation in the decaying laser plasma, as well as the intensification of the formation of large particles as a result of the aggregation of smaller ones with the increasing of the number of laser pulses. Analysis of data obtained by energy-dispersive X-ray spectroscopy (EDS) showed a significant increase of oxygen content in the irradiated steel samples, which is most pronounced in the region of regular grain structure formation.

For investigation of oxidation and surface structuring of high quality structural carbon steel (0.42 wt.% C, 0.5 wt.% Mn, 0.25 wt.% Cr) samples were processed by nanosecond pulsed laser irradiation ($\lambda = 1064$ nm, $\tau = 1$ ns, $f = 100 \div 500$ Hz, $q = 3.2 \cdot 10^9 \div 4.4 \cdot 10^9$ W/cm², $d = 80 \div 100$ μ m) at different ambient conditions. Laser treatments were carried out in air, water, 3% hydrogen peroxide (H₂O₂) and ethanol (C₂H₅OH).

Significant differences were found between laser-induced structures on steel surface in ethanol, air and peroxide (Fig. 2). On the surface of steel samples irradiated in the ambient air, wave structures with a period of 1 μ m, irregular structures with a size of 20 \div 200 nm, grains with the size of 50 \div 200 nm, pores and irregular threadlike structures were observed (Fig. 2, a, b). Quasiregular structures with a size of 100 \div 200 nm and plurality of pores with a size of 40 \div 600 nm were discovered on the surface of steel samples irradiated in the ethanol (Fig. 2, c, d). On the surface of the samples irradiated in the environment of hydrogen peroxide numerous cracks and grain structures with the size of 30 \div 70 nm were observed (Fig. 2, e, f).



air (a, b), ethanol (c, d), 3% hydrogen peroxide (e, f)
 Fig. 2. SEM image of steel irradiated in various ambiances

Analysis of data obtained by energy-dispersive X-ray spectroscopy (EDS) showed a significant increase of oxygen content in the surface layer of samples irradiated in air and hydrogen peroxide (Table 2). Besides, a significant increase of carbon content was observed for the steel sample irradiated in the air. Investigation of adhesion properties of irradiated surface, indicated that laser treatment of steel samples in air and hydrogen peroxide environments led to increasing wettability of the surface, which is expressed in a decrease of the wetting angle of 2.3 times.

Table 2. Averaged values of increasing the content of oxygen and carbon in the irradiated samples

	ethanol	3% hydrogen peroxide	air
Oxygen	1.3 times	4.4 times	5.5 times
Carbon	2.0 times	1.7 times	3.9 times

The results show a significant role of plasma formation and the surrounding environment on surface structuring and oxidation of steel, as well as the ability to control a size of the grain structures and the level of steel oxidation by changing a number of laser pulses and an environment in which the laser processing is carried out.

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