

Materials Modification by Compression Plasma Flows

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Abstract – The main principles and regularities of materials treatment by compression plasma flows generated in quasi-stationary plasma accelerators are discussed in this work. Three main directions of the CPF application are described: direct treatment of materials, mixing of a “coating-substrate” system, thin film deposition and surface layer alloying by an additional component injected into a plasma flow.

1. Introduction

Pulse plasma beams are widely used for the modification of construction and instrumental materials properties [1–3]. Compression plasma flows (CPF) generated in a quasi-stationary plasma accelerator of compact geometry [4] can be also effectively used for this purpose due to a comparatively long discharge time ($\sim 100 \mu\text{s}$), a high temperature (1–3 eV) and a high velocity of plasma particles ($(4-7) \cdot 10^6 \text{ cm/s}$). The impact of compression plasma flows with the material surface can be accompanied by the following processes [5, 6]: ablation, melting of a surface layer, liquid phase mixing under temperature and pressure gradients (in case of nonuniform element composition along the depth); rapid cooling and crystallization of a modified layer. That is why three main directions of the CPF application can be marked out: direct treatment of materials, mixing of a “coating-substrate” system, thin film deposition and surface layer alloying by an additional component injected into a plasma flow. These three directions are described below.

2. Direct plasma treatment of materials

Direct treatment of materials allows to modify the surface layer properties mainly due to the following effects: a synthesis of compounds containing elements of a plasma forming gas (e.g., nitrides), a change of grains size and morphology, initial phases modification, formation of a developed defect structure as a result of a high cooling rate.

Complicated processes taking place during the interaction of CPF with the material surface results in a substantial modification of surface morphology. CPF treatment of silicon and aluminium resulted in the formation of submicron ordered cylindrical (Fig. 1) and plate-like structures (Fig. 2) [4, 7].

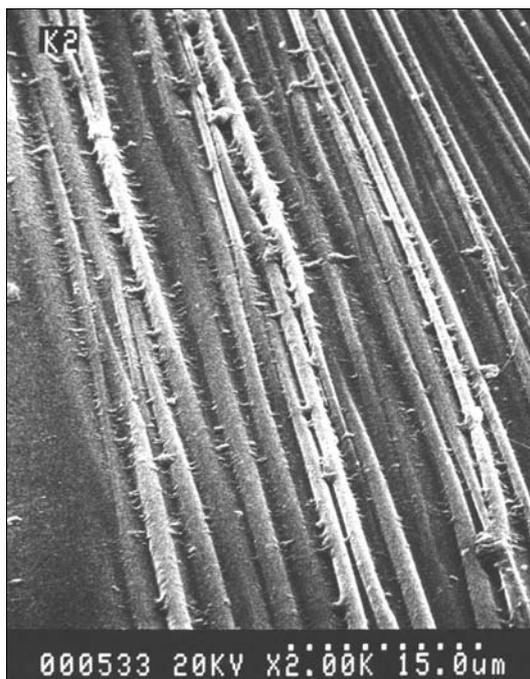


Fig. 1. Surface morphology of silicon treated by CPF

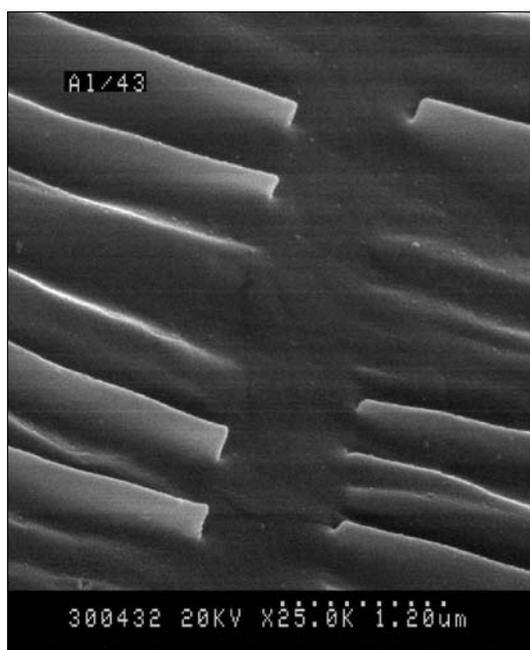


Fig. 2. Surface morphology of aluminium treated by CPF

A cellular structure can also be observed on the metals surface which is usually formed under conditions of a high-cooling speed and the impurities presence [7, 8].

Treatment of materials in nitrogen atmosphere results in nitrogen diffusion into the surface layer. One can find a nitrogen solid solution on basis of α -Fe, γ -Fe and γ -Fe₄N, ϵ -Fe_{2+x}N nitrides in the nitrogen diffusion layer after CPF treatment of iron and steels [9, 10]. AlN was found in the aluminium surface layer after treatment [11].

The modified iron layer consists of three main zones (Fig. 3) [12].

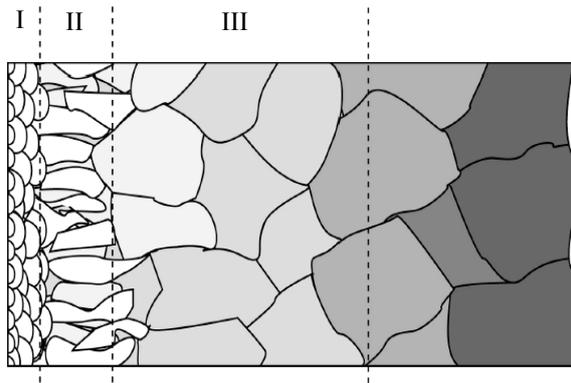


Fig. 3. Microstructure of the iron surface layer treated by CPF

The first zone with the thickness of $\sim 6 \mu\text{m}$ contains disperse grains crystallized from the melt with a high cooling speed ($\sim 10^7 \text{ K/s}$). It can contain α -Fe(N, C), γ -Fe(N, C) and γ -Fe₄N phases. The thickness of this zone increases with the increase of the energy deposited in the surface layer Q . The grains size diminishes with the growth of the number of pulses. The second zone with the thickness of $\sim 15 \mu\text{m}$ has columnar grains. Its thickness does not depend significantly on the energy deposited in the surface layer. Martensite packets were found in this zone. In case of treatment in hydrogen atmosphere the second zone is absent. The third zone is a transition layer with the thickness of a few tens of μm .

The interaction of CPF with steels is accompanied by dissolution of hardening carbides. This effect strongly influences strength properties of high-alloyed steels [10]. Treatment of AISI M2 high-speed steel resulted in partial dissolution of Fe₃W₃C and VC carbides (Fig. 4), thus leading to the surface layer hardness decrease.

The increase of energy deposited in the surface layer resulted in the increase of iron and low-alloyed steels hardness and in the decrease of high-alloyed steels hardness [10, 12]. CPF treatment also resulted in the decrease of the friction coefficient for all investigated types of steel [10, 12]. For a high-alloyed steel, it is connected with a large volume fraction of residual austenite [10].

3. Mixing of a “coating-substrate” system

CPF can also be effectively used for “coating-substrate” system mixing, thus leading to alloying of a substrate with atoms of coatings and a plasma forming gas. An alloying element (an element of the coating) is uniformly distributed along the mixed layer after treatment with a few plasma pulses (Fig. 5). The thickness of the mixed layer depends on a treated material. It amounts to ~ 10 – $15 \mu\text{m}$ in steels [5, 13] and up to $70 \mu\text{m}$ in aluminium alloys [14].

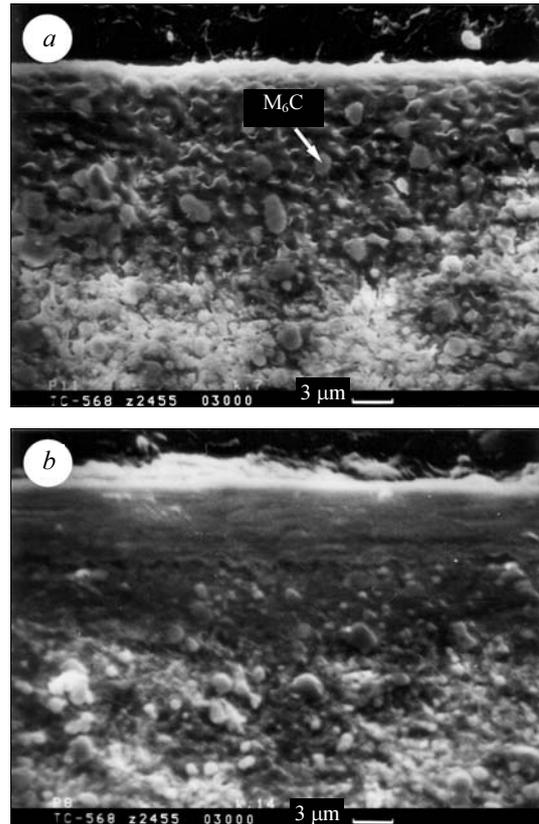


Fig. 4. Morphology of AISI M2 steel cross section after one pulse of CPF treatment with $Q = 5$ (a) and 10 J/cm^2 (b)

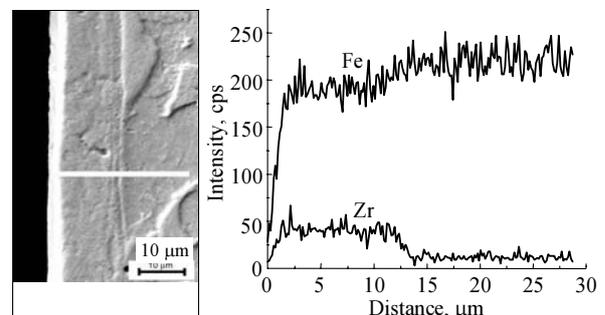


Fig. 5. Cross section morphology and distribution of elements along the depth in the sample of “Zr-steel” system treated by 5 pulses CPF at $Q = 13 \text{ J/cm}^2$ per pulse

The structure of the modified layer is the following [15]. The first layer with the thickness of $\sim 1 \mu\text{m}$ can contain nitrides (in case of treatment in nitrogen

atmosphere). Preliminary deposition of elements like Ti and Zr with low formation enthalpy of nitrides allows forming TiN or ZrN in this layer (Fig. 6). The second layer (almost the whole mixed layer) contains elements of a coating and a substrate. The third layer (a thermal influence zone) with the thickness comparable with the thickness of the mixed layer has a modified structure of the substrate.

The phase composition of the mixed layer is strongly dependent on the concentration of the alloying elements. Phase equilibrium diagrams can be used for preliminary determination of the phase composition providing that the element composition of the mixed layer is known or can be estimated [16].

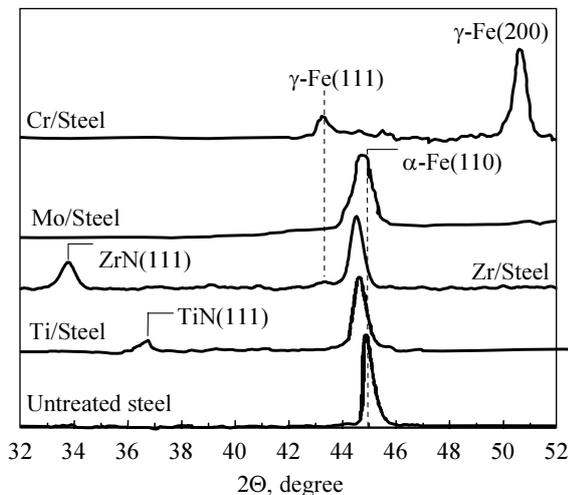


Fig. 6. X-rays diffraction patterns of an untreated steel sample and samples of a "coating-substrate" system after 5 pulses of CPF treatment (1 pulse for Ti/steel) at $Q = 13 \text{ J/cm}^2$ per pulse

The element composition can be controlled in the following ways. For the first, it can be changed by the variation of treatment parameters: the number of pulses and the energy transferred to a sample. After one pulse of treatment the average concentration of the alloying element \bar{c} can be roughly estimated (without taking into account ablation) as $\bar{c} = t_{coat} / t_{mix}$, where t_{coat} is the thickness of the preliminary deposited coating and t_{mix} is the thickness of the modified layer.

The previous experiments showed that the concentration of the alloying element did not exceed ~15 at.% after a few plasma pulses necessary to achieve uniform elements distribution in the mixed layer [13–17].

Another way of the element concentration change is a variation of a coating thickness. For example, the change of Ti coating thickness from 0.5 to 5.0 μm resulted in the variation of Ti concentration in the mixed layer from 3 to 31 at % after 3 pulses of CPF treatment. This led to changes in the phase composition: supersaturated solid solution $\alpha\text{-Fe}(\text{Ti}) \rightarrow \alpha\text{-Fe}(\text{Ti}) + \text{Fe}_2\text{Ti} + \text{TiN}$ (Fig. 7). Similar result can be achieved

by multiple repetition of titanium coating deposition processes and CPF processing.

The mixed layer possesses enhanced tribological properties. Alloying of a low-alloyed carbon steel leads to the 2–3 times increase of its hardness up to 6.5 GPa and to the decrease of its friction coefficient to 0.1 [13, 15]. Alloying of the surface layer with refractory materials allows avoiding the hardness decrease in high-alloyed steels [12] and allows increasing thermal stability of steels properties [18, 19]. CPF treatment can also be used for semiconductors properties changes.

For example, alloying of silicon with iron atoms leads to $\alpha\text{-FeSi}_2$ formation in the surface layer, thus changing its electrical properties [20].

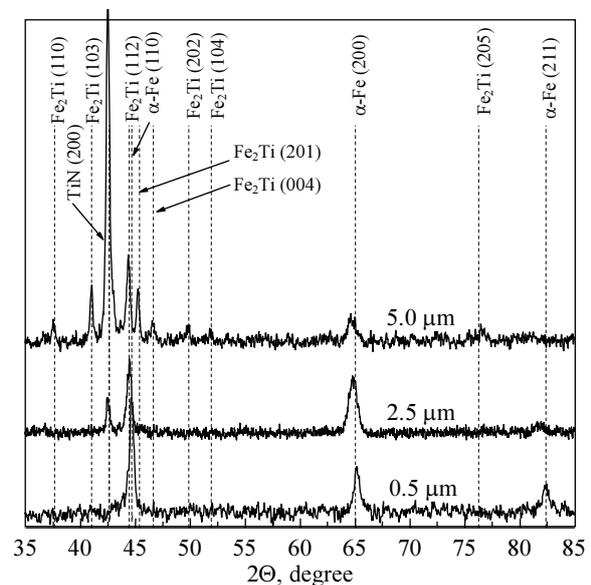


Fig. 7. X-rays diffraction patterns of "Ti-steel 3" system samples with a different thickness of the Ti coating after CPF treatment with $Q = 13 \text{ J/cm}^2$ per pulse

4. Injection of an additional component into a plasma flow

Alloying of the surface layer can be also carried out by the injection of an additional component into a plasma flow. The findings showed that the injection of a metal powder into a plasma flow led to the deposition of a metal containing a thin film on the silicon surface [21]. The film consists of a spherical particles monolayer, up to 200 nm in size, bounded to each other (Fig. 8).

These particles cover both plane surface areas and cylindrical silicon structures formed by plasma treatment [4]. Spherical particles deposition takes place from the area of a shock-compressed layer after plasma shell disintegration. The shock-compressed plasma layer is formed during the impact of CPF with the surface throughout the discharge duration. It consists of target material ablation products and elements of the plasma flow. Clusterization of a cooling vapor

in the near surface layer and particles (clusters) deposition on the crystallized surface take place after plasma shell disintegration.

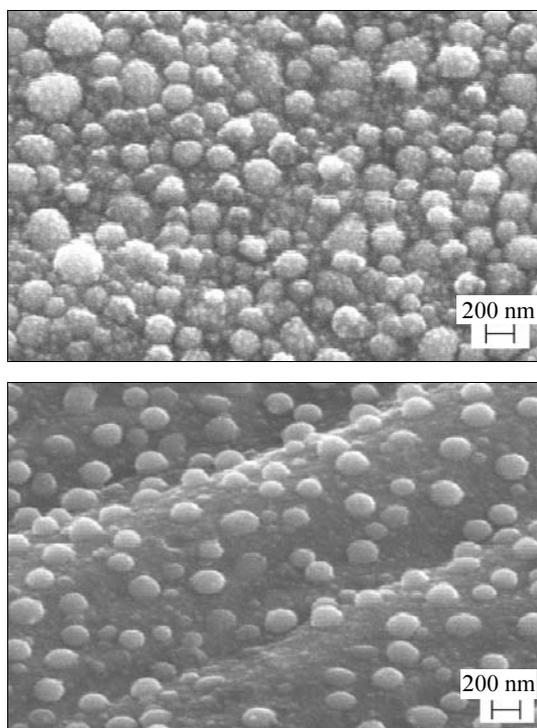


Fig. 8. Morphology of the silicon surface treated by one pulse of CPF ($Q = 15 \text{ J/cm}^2$) with different concentrations of a metal component in a plasma flow

Treatment of a carbon steel by compression plasma flows with an injected tungsten powder also resulted in the formation of a tungsten containing thin film consisting of clusters with the size of 100–200 nm [22]. The increase of the pulse number led to the liquid phase mixing of the earlier deposited film with the substrate material, thus resulting in deeper penetration of film elements into the bulk and in the formation of the steel surface layer alloyed with tungsten. Treatment resulted in the formation of $\text{Fe}_3\text{W}_3\text{C}$ and WC carbides in the surface layer, thus leading to the increase of hardness up to 8 GPa [22].

5. Conclusions

Generation of compression plasma flows is a promising technique for materials modification. It allows forming different submicron structures that are of interest for surface science.

Compression plasma flows provide the formation of a surface layer with improved properties the thickness of which can vary from micrometers to tens of micrometers.

Different methods of alloying by compression plasma flows allow using it for the predictable formation of a variety of alloys and compounds in the sur-

face layer. A high cooling speed gives a possibility of metastable phases formation.

Thus, compression plasma flows can also be used as a new materials formation technique.

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