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Cleaning of steel surface from scale by compression plasma flows

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ABSTRACT

The investigation of compression plasma flow treatment parameter effect (the number of pulses and the energy absorbed by the surface layer) on cleaning efficiency of the steel surface from scale ($\text{Fe}_2\text{O}_3/\text{Fe}_3\text{O}_4/\text{FeO}$) is the main aim of the research carried out in this work. The results of the phase and element composition, cross-section morphology investigations are presented. The findings showed that efficiency of plasma cleaning increased with the growth of the pulses number (1–3) and the energy absorbed by the surface layer (10–20 J/cm² per pulse). Evaporation and cracking of scale due to the difference in coefficients of linear expansion of Fe_2O_3 , Fe_3O_4 , FeO and steel are supposed to be the main reasons for scale removal by plasma flow impact.

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1. Introduction

Different types of surface treatment techniques based on the impact of laser, cluster and plasma beams can be used for cleaning the surface from contaminants, oxides, coatings, etc. [1–10]. Generation of shock waves [5,6], evaporation and ablation [5], difference in coefficients of linear expansion [7], physical sputtering [8] and etching [8–10] are the main mechanisms that are considered to be responsible for cleaning depending on a beam type and its energy. The impact of high-power ($\geq 10^6$ W/cm²) pulsed electron, ion and plasma beams on materials results in ablation and rapid heating of the surface layer [11–16]. These processes can also be applied for surface cleaning or removal of surface layers.

Earlier it was found that treatment by compression plasma flows (CPF) could be used for refinement and homogenization of surface layer structure, alloying by additional elements thus resulting in substantial improvement of steels mechanical and tribological properties, their thermal stability [17–19]. The findings also showed that CPF impact led to removal of oxide layers from the surface of oxidized samples [20]. Such a cleaning process can be considered as a preliminary operation in the technological process of metal and alloy surface layer modification by CPF.

Short treatment time (10^{-4} s) in comparison with traditional ion-plasma sputtering, lower power inputs (in comparison with continuous ion-plasma treatment) and a possibility of operation at normal pressure (without a vacuum system) are the main advantages of such a type of cleaning technique. The main disadvantages consist of the following:

only a flat surface can be effectively treated, plasma flows are less controllable than ion or electron beams, a comparatively small treatment area (~ 20 cm² per pulse).

As a cleaning technique this type of treatment may be used for removal of scale from the surface of thin steel sheet products that cannot be subject to long thermal treatment because of tension and subsequent bending. The investigation of CPF treatment parameter effect (the number of pulses and the energy absorbed by the surface layer) on cleaning efficiency of the steel surface from artificially grown scale was the main aim of the research carried out in this work.

2. Experimental

The samples used ($\varnothing 15$ mm) were made of a carbon steel (0.3 C; 0.2 Si, 0.5 Mn wt.%, Fe-balance). The scale formed on steel sheet products with the thickness of the order 10^1 – 10^3 μm can contain layers of Fe_2O_3 , Fe_3O_4 and FeO. That is why to simulate phase composition of the steel scale the temperature of annealing was chosen higher than 843 K (the temperature necessary to form FeO [21]) but less than 1000 K to avoid polymorphic transformation bcc \rightarrow fcc. The scale layer was formed on the steel sample surface by annealing at 973 K in air for 3 h. Annealing for more than 4 h resulted in scale layer delamination. The samples were cooled in the furnace after annealing. The grown scale must contain $\text{Fe}_2\text{O}_3/\text{Fe}_3\text{O}_4/\text{FeO}$ (adjacent to steel) layers according to Ref. [21].

CPF were obtained using a gas-discharge magneto-plasma compressor of compact geometry. CPF treatment was performed in a “residual gas” mode in which the vacuum chamber was filled with nitrogen up to the preset pressure of 400 Pa. The discharge device and principles of CPF generation were described in detail earlier [22]. The plasma flow parameters were as follows: pulse duration ~ 100 μs , flow velocity $(5 \div 6) \cdot 10^6$ cm/s and electron concentration $(4 \div 7) 10^{17}$ cm⁻³.

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Treatment of the oxidized samples was carried out by 1–3 pulses. Pulse-repetition frequency was 0.05 Hz. The flow diameter in the area of sample location was 4–5 cm. The energy density absorbed by the surface layer (Q) was 10 J/cm^2 and 20 J/cm^2 per pulse according to calorimetric measurements.

Element composition was analyzed by means of the non-Rutherford backscattering spectrometry (nRBS) of He^+ ions with an energy of 6 MeV. A high energy of helium ions allowed to increase the yield of ions scattered on light impurities due to resonance. Non-Rutherford cross sections were used for calculation by SIMNRA code [23]. The High Voltage Engineering tandemron system accelerator (Augsburg University, Germany) was used. Phase composition of the samples was investigated by the X-ray diffraction analysis (XRD) in Bragg–Brentano geometry in $\text{Cu K}\alpha$ radiation using a DRON 4–13 diffractometer. Cross-section morphology as well as element composition were analyzed by means of scanning electron microscopy (SEM) using a LEO1455VP device equipped with an energy-dispersive X-ray Röntec detector.

3. Results and discussion

In order to analyze the reasons for scale layer removal during CPF impact the structure, phase and element composition of an artificially grown scale layer were investigated. The XRD analysis showed that the analyzed layer with a thickness of $\leq 4 \mu\text{m}$ (this is an X-ray penetration depth if one assumes 75% of radiation absorption) mainly consisted of Fe_2O_3 oxide (Fig. 1). At the same time some diffraction lines could be attributed to Fe_3O_4 phase due to a small difference in interplanar spacings. nRBS investigations confirmed that the surface oxide layer (the analyzed depth $< 3 \mu\text{m}$) was Fe_2O_3 (Fig. 2). The data of SEM showed that the thickness of the oxide layer was $27 \mu\text{m}$ and it had two main sublayers with a different concentration of oxygen (Fig. 3). The upper sublayer was enriched with oxygen. According to the data of XRD and nRBS it can be characterized as Fe_2O_3 . The upper sublayer contained a lot of pores. The lower sublayer was enriched with iron. It looked lighter due to the element contrast in back-scattered electrons during SEM investigations (Fig. 3a). Oxidation kinetics in this experiment must correspond to the laws of conventional atmospheric corrosion. Due to oxygen diffusion in the oxide layer out of atmosphere to the steel surface the upper layer of scale must contain more oxygen than that located near the steel surface, which is consistent with the experimental data

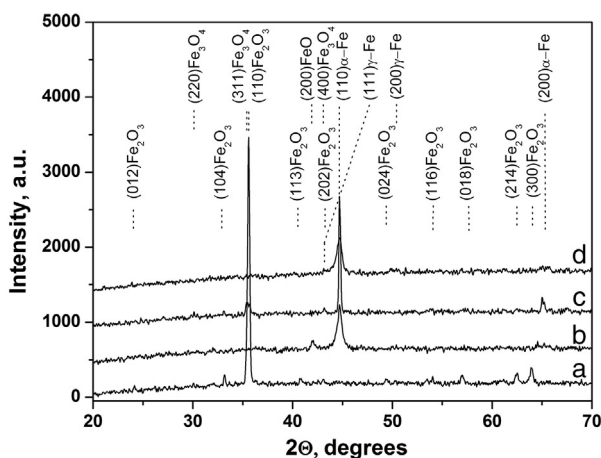


Fig. 1. XRD patterns of the oxidized steel samples before (a) and after CPF treatment: 10 J/cm^2 and 3 pulses (b), 20 J/cm^2 and 1 pulse (c), 20 J/cm^2 and 3 pulses (d). Diffraction peaks were indicated according to PC Powder Diffraction Files, JCPDS-ICDD: Fe_2O_3 – PDF# 79–1741, Fe_3O_4 – PDF# 82–1533, FeO – PDF# 06–0615, $\alpha\text{-Fe}$ – PDF# 06–0696, $\gamma\text{-Fe}$ – PDF# 23–0298.

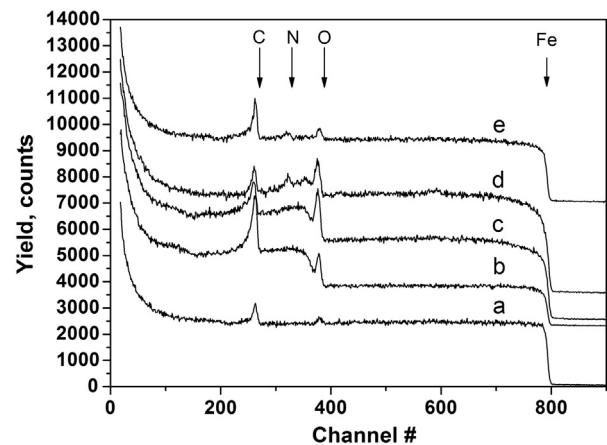


Fig. 2. nRBS spectra of the steel sample (a) and oxidized steel samples before (b) and after CPF treatment: 10 J/cm^2 and 1 pulse (c), 10 J/cm^2 and 3 pulses (d), 20 J/cm^2 and 3 pulses (e).

(Fig. 3b). Oxidation occurs in comparatively equilibrium conditions, that is why the lower sublayer can contain Fe_3O_4 and FeO [21].

CPF treatment of the oxide/steel system with one pulse at $Q = 10 \text{ J/cm}^2$ led to a homogeneous decrease of the oxide thickness up to $22 \mu\text{m}$ (Fig. 4a). Rare cracks could be found in the oxide layer. Evaporation or ablation of the surface layer may be the mechanism of oxide removal at this treatment regime. According to the nRBS data (Fig. 2) the Fe:O ratio in the analyzed layer was close to 1:1 allowing to suppose FeO formation. At the same time XRD analysis did not reveal any changes in phase composition of the oxide surface layer in comparison with the untreated one. The increase of the number of pulses up to three did not provide a full removal of the scale layer (Fig. 4b). Local oxide spots with a thickness of $\sim 5 \mu\text{m}$ were present on the steel surface. A direct plasma interaction with steel took place at this treatment regime in areas where the oxide layer was eliminated by the previous pulses. The thickness of the melted steel surface layer was $\sim 5\text{--}7 \mu\text{m}$,

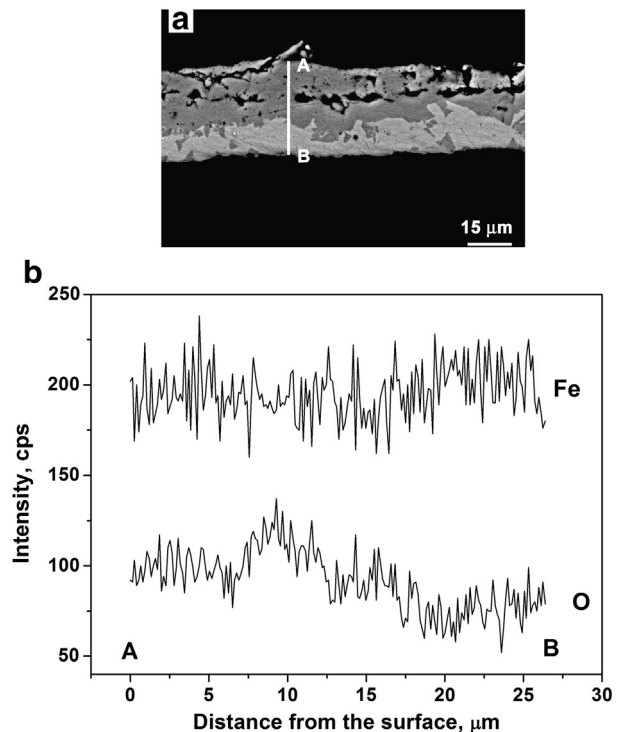


Fig. 3. Cross-section morphology of the oxide layer (a) and distribution of O and Fe along the line A–B across the oxide layer (b).

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