ALLOYING AND MODIFICATION OF STAINLESS STEELS BY POWERFUL PLASMA STREAMS

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The stainless steel surfaces coated of tungsten have been alloyed and modified with powerful QSPA Kh-50 plasma streams. The plasma streams exposures result in modification of steel-based materials and formation of re-solidified layers. The changes of substrate texture were also registered. Phase characterized by body-centered cubic lattice appeared due to recrystallization of affected material. Thus, the favorable conditions were created for penetration of tungsten into the stainless steel bulk. Growth of lattice parameter was observed as result of plasma irradiation of coated samples. It's indication of tungsten penetration into the depth of substrates.

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INTRODUCTION

Reduced-activation steels are considered now as structure materials for nuclear and fusion devices due to their high swelling resistance and low irradiation creep rate (the slow change in dimensions of a material due to prolonged stresses, caused by X-rays, γ-rays and neutron irradiation, amongst others). In particular, those steels could be a possible option for plasma-facing material of DEMO first wall surfaces [1-3].

The main drawback of steels for their application as plasma facing materials is high sputtering rate under influence of energetic ions (particularly hydrogen isotopes). It is directly related to impurity generation as well as to the lifetime of plasma- facing components [3, 4]. One of the promising ways for improvement of steel properties is alloying of their surface layer with refractory elements [3]. For example, plasma species can be considered as a source of alloying elements to be introduced into a modified layer structure [5]. Another possibility of alloying under the pulsed plasma processing is mixing of previously deposited thin ($h_{coat} < h_{melt}$) coatings of different predetermined composition with the substrate in the course of melting driven by powerful plasma impacts [6, 7].

Alloying of surface layer in result of the coating-substrate mixing allows achievement of desirable chemical composition in surface layers [5-9]. Nevertheless, modification processes in plasma facing materials that induced by repetitive plasma impacts and synergetic effects from different influencing factors are still not understood well to make reasonable predictions for steels behaviour during the reactor operation. All mentioned issues require comprehensive studies of prospective steel materials with different powerful plasma devices-simulators and coordinated activities that will address relevant issues associated with the impact of intense heat and particle loads on plasma facing materials.

Analysis and comparison of experimental results on materials behavior under irradiation with steady-state and high-power pulsed plasma streams generated in different plasma devices with various specific heat loads and different particle fluxes gives the unique possibility to understand an important common features of surface damage in extreme conditions, to investigate the peculiarities of surface modification for reduced activation steels and possible erosion mechanisms in dependence on the plasma parameters and the impacting load.

1. EXPERIMENTAL DEVICES AND DIAGNOSTICS

Samples of Cr18Ni10Ti (analog of stainless steel SS 321) austenitic steel used in experiments were 20 × 20 × 1 mm. High quality tungsten coatings were created using PVD technique [7] in a Bulat type facility. Coatings with thickness of about 3 μm were deposited during 3 min in argon of $p = (4…5)\times10^{-4}$ Torr. Parameters of arc discharge are as follows: current of arc $I_{arc} = 230$ A and biasing voltage of $U_{bias} = 140$ V. Before deposition of coatings, ionic cleaning of the surface (etching duration 2 min, $U_{bias} = 1.5$ kV and $I_{arc} = 100$ A) was applied.

Surface modification by powerful pulsed plasma treatment was carried out with the use of the quasi-stationary plasma accelerator QSPA Kh-50 [10]. Main parameters of QSPA plasma streams are as follows: ion impact energy was about (0.4…0.6) keV, the maximum plasma pressure up to 0.32 MPa, and the plasma stream diameter of 18 cm. The power load shape is approximately triangular, and the pulse duration of 0.25 ms [10-12].

The energy density in free plasma and surface heat load were measured by the local calorimeters. Surface analysis of exposed samples was carried out with an optical microscope MMR-4, equipped with CCD camera. Measurements of weight losses and precise measurements of the surface roughness with the Hommelwerke tester T500 were also performed.

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X-ray diffraction (XRD) has been used to study structure, sub- structure and stress state of targets. ϑ-2ϑ scans were performed using a monochromatic Cu-K_α radiation [11-13]. Computer processing of the experimental diffraction patterns was performed using the new profile 3.5 software package [12]. Comprehensive analysis of diffraction peaks intensity, profiles, width (B), angular positions was applied to evaluate texture, coherent scattering region size [12]. Changes of phase state on the surface were evaluated from XRD spectrum analysis. Residual macro-stresses (σ) and the lattice parameter in the unstrained state (a_0) were determined using a- $\sin^2\psi$-plots [11, 12].

2. EXPERIMENTAL RESULTS

The initial microhardness and roughness of Cr18Ni10Ti samples were 350 kg/mm^2 and R_a < 0.1 μm (R_{max}≈ 0.1 μm) respectively (Fig. 1). Only lines of γ-Fe phase are observed on the initial surfaces (Fig. 2,a). Lattice spacing in stress free state is measured as $a_{\gamma\text{-Fe}}$ ≈ 0.359 nm. It should be mentioned that face centered cubic lattice is attributed to γ-Fe.

The cycle of steel alloying consisted of two stages. During first stage the tungsten coating was deposited on the surface by PVD method. The surface roughness is slightly increased after deposition of tungsten coatings (R_a < 0.1 μm, R_{max} ≈0.9 μm, Fig. 1). At the second stage the coated samples were processed with hydrogen plasma streams in QSPA Kh-50 device.

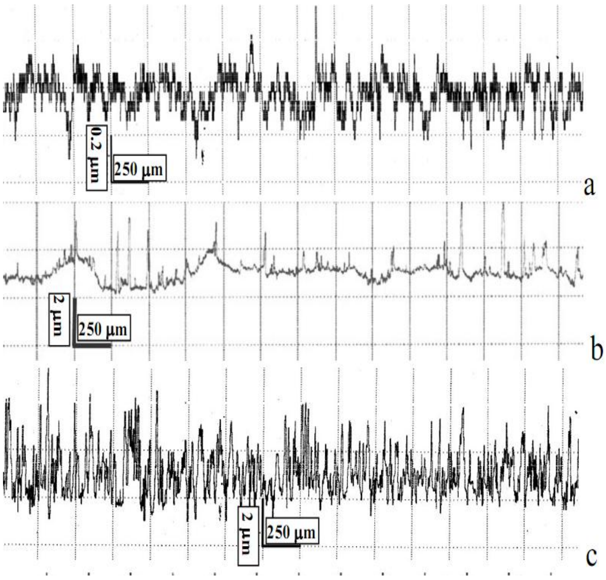

Fig. 1. Profiles of sample surface: initial (a) and after W coating deposition (b), modification by 5 plasma pulses of 0.6 MJ/m^2 (c)

In our previous studies of tungsten material the melting (0.6 MJ/m^2) and cracking (0.3 MJ/m^2) thresholds have been determined for QSPA plasma pulses. The evaporation onset is estimated as 1.1 MJ/m^2 [10, 14]. As result of plasma irradiation of surfaces coated by tungsten with heat load below the melting threshold a mesh of major cracks appears upon the exposed target surface. Only few particles were ejected from the exposed surface as registered by CCD imaging. In the case of plasma heat loads resulted in pronounced surface melting the droplets emission became dominant [14]. For stainless steel the melting threshold is experimentally evaluated as 0.4…0.5 MJ/m^2 for applied QSPA plasma pulses. From this reason, the heat load to the coated target surface was chosen to be on the level of 0.6 MJ/m^2 (i.e., in the vicinity of the tungsten melting threshold).

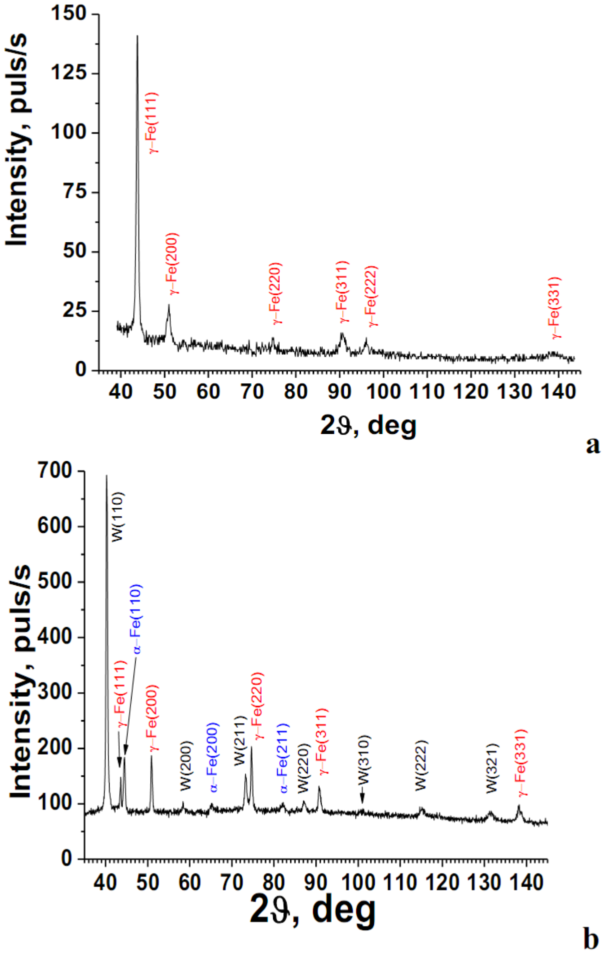

Fig. 2. Diffraction patterns (Cu-K_α radiation) of stainless steel sample: initial state (a) and after deposition of tungsten and irradiation of 5 plasma pulses of 0.6 MJ/m^2 (b)

After irradiation of the sample surface with 5 plasma pulses of above mentioned heat load molten and further re-solidified surface layer is appeared (Figs. 3, 4). The surface morphology is developed also due to generation of both macro and micro cracks [15]. The roughness of exposed surfaces increased up to R_a ≈0.1 μm, R_{max} ≈1.3 μm (see Fig. 1,c.). The delamination of coatings was not observed (see Fig. 4). Microhardness of modified surface was increased up to 400 kg/mm^2 due to the stresses and quenching introduced by plasma treatment [16].

α-Fe phase is recognized together with lines of γ-Fe phase and W on the treated surfaces. Intensity of tungsten lines essentially exceeds the intensity of substrate lines. The intensity of γ-Fe lines increased also. It indicates a re-crystallisation and improvement structure of exposed surfaces (Fig. 2,b). Both α-Fe and tungsten are characterized by body-centered cubic crystal structure. Therefore, presence of α-Fe phase creates helpful conditions of tungsten penetration into affected layer. It agrees with increasing of lattice spacing of γ-Fe from 0.359 nm till $a_{\gamma\text{-Fe}}$= 0.35943 nm. Such increase of a_0 can be explained by increasing tungsten concentration up to 2 atomic percent in modified subsurface layer [13].

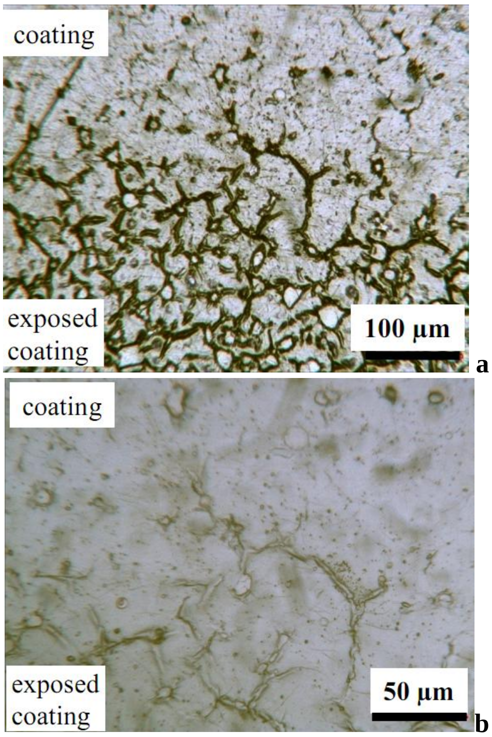

Fig. 3. Images of exposed area edge with different magnification

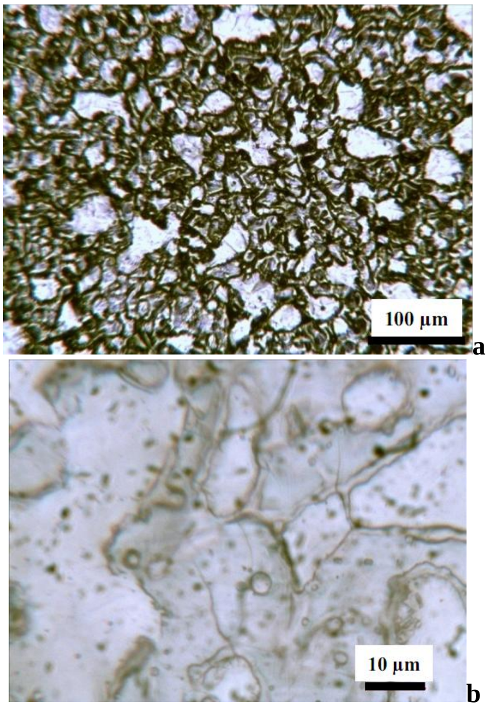

Fig. 4. Images of exposed area with different magnification

Depth of tungsten penetration was estimated by means of measurements of intensity of different lines (W, α-Fe, γ-Fe) in exposed and unexposed sides of sample. As it was found, tungsten is concentrated near surface layer of 0.38 μm.

It should be mentioned that measured value of $a_{\gamma\text{-Fe}}$ is less than the γ-Fe reference lattice spacing of 0.3637 nm. For tungsten (0.31637 nm) and α-Fe (0.28596 nm) lines, the lattice spacing also some less than corresponding reference values (a_W =0.3165 nm and $a_{\alpha\text{-Fe}}$ = 0.2866 nm) due to available surplus of vacancies in the modified layer (Fig. 5). Symmetrical tensile stresses of 269 MPa and about 880 MPa registered from α-Fe and γ-Fe lines, respectively. Tensile stresses of 218 MPa registered from tungsten line.

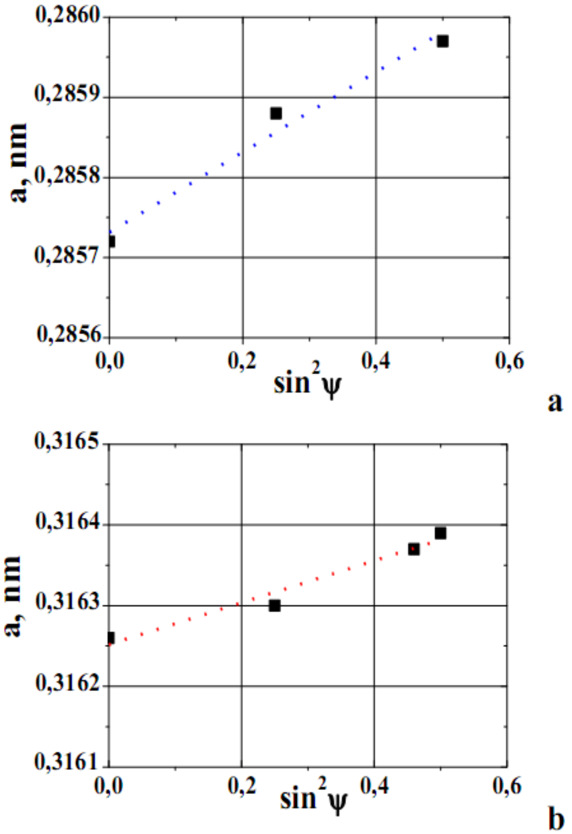

Fig. 5. a -sin$^2\psi$*-plots from tungsten (a) and α-Fe (b) line for exposed sample*

CONCLUSIONS

Experimental studies of surface modification of stainless steel samples covered by tungsten coatings have been performed with a quasi-stationary plasma accelerator QSPA Kh-50. Plasma heat load on the surface was about 0.6 MJ/m^2 (i.e., near tungsten melting threshold).

Therefore, melted, re-solidified layer is developed on exposed surfaces. Modified layer included mixed coating material with substrate. α-Fe phase is recognized together with lines of γ-Fe phase and tungsten on treated surfaces. Lattice spacing of γ-Fe in a stress free state is increased from 0.359 nm up to $a_{\gamma\text{-Fe}}$= 0.35943 nm. The concentration of tungsten is amounted to 2 atomic percent in modified subsurface layer. Estimated depth of tungsten penetration into the modified layer of steel sample achieved 0.38 µm.

The surface morphology is developed mostly by melting and re-solidification processes in the course of plasma treatment. Both major and micro cracks are appeared on the exposed surfaces. However, any delamination of coatings was not observed.

ACKNOWLEDGEMENTS

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ЛЕГИРОВАНИЕ И МОДИФИКАЦИЯ НЕРЖАВЕЮЩЕЙ СТАЛИ МОЩНЫМИ ПОТОКАМИ ПЛАЗМЫ

В.А. Махлай, Н.Н. Аксёнов, О.В. Бырка, А.Г. Чунадра, С.С. Геращенко, С.В. Малыхин, И.Ф. Михайлов, К.Н. Середа, С.В. Суровицкий

Поверхности из нержавеющей стали с покрытием из вольфрама были легированы и модифицированы мощными плазменными потоками в КСПУ Х-50. Облучение потоком плазмы приводит к модификации материалов на основе сталей и формирование повторно затвердевшего слоя. Изменения текстуры подложки были также зарегистрированы. Фаза, которая характеризуется объёмно центрированной кубической решёткой, появилась вследствие рекристаллизации облучённого материала. Таким образом, были созданы необходимые условия для проникновения вольфрама в нержавеющую сталь. Рост параметра решётки наблюдался в результате плазменного облучения образцов с покрытием. Это указывает на проникновение вольфрама в глубину субстратов.

ЛЕГУВАННЯ І МОДИФІКАЦІЯ НЕРЖАВІЮЧОЇ СТАЛІ ПОТУЖНИМИ ПОТОКАМИ ПЛАЗМИ

В.А. Махлай, М.М. Аксьонов, О.В. Бирка, А.Г. Чунадра, С.С. Геращенко, С.В. Малихін, І.Ф. Михайлов, К.М. Середа, С.В. Суровицький

Поверхні з нержавіючої сталі з покриттям з вольфраму були леговані і модифіковані потужними плазмовими потоками в КСПП Х-50. Опромінення потоком плазми призводить до модифікації матеріалів на основі сталей і формування повторно затверділого шару. Зміни текстури підкладки були також зареєстровані. Фаза, яка характеризується об'ємно центрованою кубічною решіткою, з'явилася внаслідок рекристалізації опроміненого матеріалу. Таким чином, були створені необхідні умови для проникнення вольфраму в нержавіючу сталь. Зростання параметра решітки спостерігався в результаті плазмового опромінення зразків з покриттям. Це вказує на проникнення вольфраму в глибину субстратів.