

# STRUCTURE EVOLUTION OF TUNGSTEN COATINGS EXPOSED TO PLASMA FLOWS UNDER ITER ELM RELEVANT CONDITIONS

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Structure, substructure and stress parameters both on the surface and distributed by depth in the tungsten/steel coatings exposed to high energy hydrogen plasma fluxes were studied by X-ray diffraction methods. The hydrogen plasma exposure results in the following recrystallization processes in the thin surface layer of the coating: appearing the texture axis [100] normal to the surface; increasing the coherence length from 60 nm in the initial state to 80 nm after the plasma exposure; completely annealed micro-strains; and dislocation density lowered twice in the exposed surface layer – all these facts confirm the thermal character of the hydrogen plasma influence. No structure variations were observed already at the depth near 1  $\mu\text{m}$  of the coating.

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## INTRODUCTION

One of the important issues determining the life-time of the components and safety of the ITER tokamak-reactor is the behavior of plasma-facing materials and components under repetitive plasma loads onto the first wall and the diverter of the fusion reactor [1].

Tungsten and tungsten alloys are primary Plasma Facing Materials (PFM) for next-step fusion devices such as ITER and DEMO due to high thermal conductivity, high temperature strength and stability, high recrystallization temperature, and high sputtering threshold for hydrogen [2-6]. Tungsten coatings on different substrates might be a less expensive alternative to the bulk tungsten for armoring the first wall in DEMO. Therefore, one of the most crucial issues is the problem of lifetime of the tungsten coatings under cyclic plasma loads relevant to edge localized modes and disruption [7-11]. The main goal of the work was studying the structure, substructure and stress parameters both on the surface and distributed by depth in tungsten coatings exposed to high energy hydrogen plasma fluxes.

## 1. SAMPLES AND INVESTIGATION TECHNIQUE

Tungsten (W) coatings (thickness of 0.6 mm) were deposited with Low Pressure Plasma Spraying technique on stainless steel plates of 50×50×3 mm. Initial surface roughness of coating is higher ( $R_{\text{max}} = 15.5 \mu\text{m}$ ;  $R_z = 12.1 \mu\text{m}$ ;  $R_a = 1.8 \mu\text{m}$ ). Features of structure, substructure and stress state of the deposited coatings were studied in initial state and after plasma irradiation.

The coatings have been irradiated by hydrogen plasma within a quasi-stationary plasma accelerator QSPA Kh-50. The main parameters of plasma flows are as following: ion energy up to 400 eV, plasma maximum pressure of 0.32 MPa. The surface plasma

loads measured with a calorimeter were 0.75 MJ/m<sup>2</sup>, i.e. between the tungsten cracking (0.3 MJ/m<sup>2</sup>) and evaporation (1.1 MJ/m<sup>2</sup>) thresholds. The plasma pulse shape is triangular with pulse duration of 0.25 ms.

X-ray diffraction (XRD) has been used to study structure, substructure and stress state of the samples.  $\vartheta \dots 2\vartheta$  scans were performed using a monochromatic Cu-K $\alpha$  radiation. For investigation of the depth structure distribution, additional measurements using grazing geometry were carried out. Computer processing of the experimental diffraction patterns was carried out using the “New profile 3.5” software package. The analysis of diffraction peaks intensity, profiles, width ( $B$ ), angular positions was applied to evaluate stress state, texture and coherent lengths. Residual macro-stresses ( $\sigma$ ) and the lattice parameter in the unstrained state ( $a_0$ ) were determined using  $a - \sin^2\psi$ -plots. Analysis of the average coherence length (related with the dislocation density in the grain boundaries) and the average micro-strain value (related with density of chaotically distributed dislocations inside the coherence domain) has been carried out by the approximation method.

## 2. RESULTS AND DISCUSSION

The X-ray diffraction patterns are shown for the coatings in the initial state (below) and after plasma exposure (up) in Fig. 1. Texture was not observed in the coatings in the initial state. Non-uniform distributions of residual stresses and the lattice parameter  $a_0$  in the sub-surface layer were also determined (Figs. 2, 3). The maximum level of residual stress of 1.6 GPa was registered in the layer of 0.07  $\mu\text{m}$ , whereas 65 MPa was observed at the depth of 1  $\mu\text{m}$ . The maximum lattice parameter of  $a_0 = 0.31646 \text{ nm}$  was determined at the depth of near 1  $\mu\text{m}$  where the stresses were minimum. It should be noted, that the obtained lattice parameter corresponds to the reference value (0.3165 nm). Closer to the surface, the  $a_0$  parameter decreased to 0.31525 nm, the fact can be explained by excess point defects (vacancies) in the tungsten lattice.

Additionally, we observed the texture with axis [100] normal to the surface after plasma irradiation. The texture occurring indicates the recrystallization process in the surface layer of the affected material. It should be noted, that the diffraction maxima from the substrate material are absent in the diffraction pattern (see Fig. 1). This indicates that the thickness of the tungsten coating is still not less than 5  $\mu\text{m}$ .

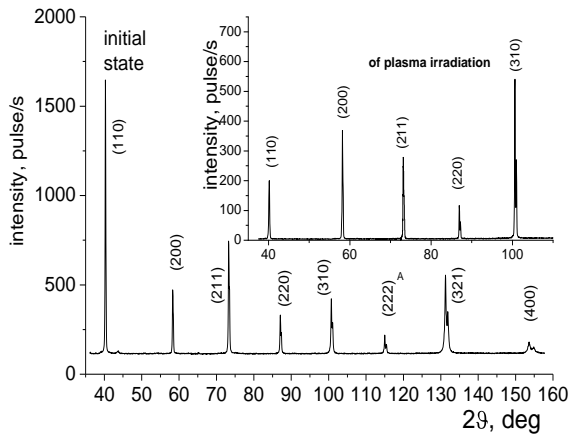


Fig. 1. X-ray diffraction patterns of a tungsten coating on steel in the initial state (below) and after plasma exposure (up)

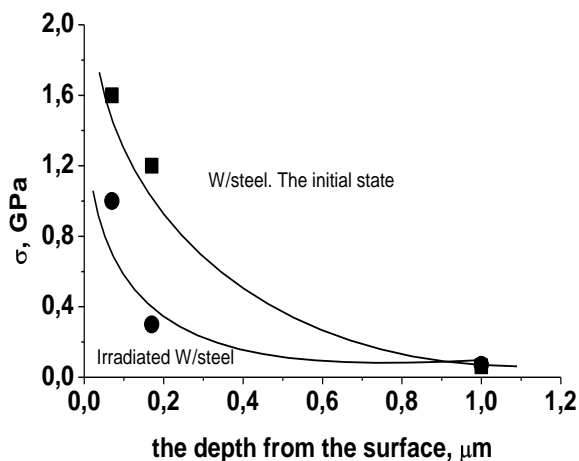


Fig. 2. Stress distribution over the depth of the W coating before and after hydrogen plasma exposure

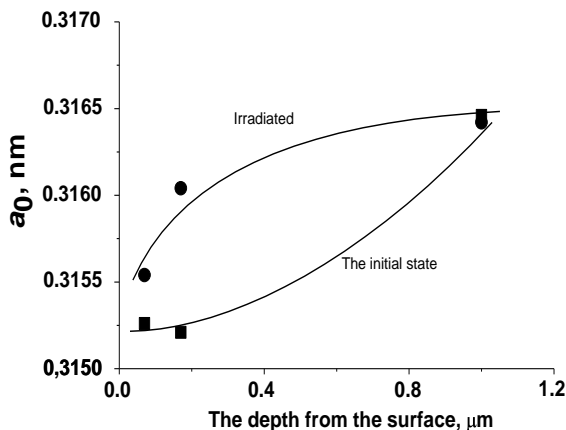


Fig. 3. Distribution of the lattice parameter over the depth of the W coating in the initial state and after hydrogen plasma exposure

Corrugated structure of hills, cracks and re-solidified large particles appears on the exposed surface and inside the craters (Fig. 4). Such structure contributes to the growth of surface roughness ( $R_{\text{max}} = 51.3 \mu\text{m}$ ;  $R_z = 45.2 \mu\text{m}$ ;  $R_a = 10.2 \mu\text{m}$ ). Plasma impacts cause the melting/dust particles splashing from the exposed surface (Fig. 5). Their maximum velocity is below 15 m/s. The particles weakly connected with the surface are able to be melted and ejected from surface under heat loads even below the melting threshold.

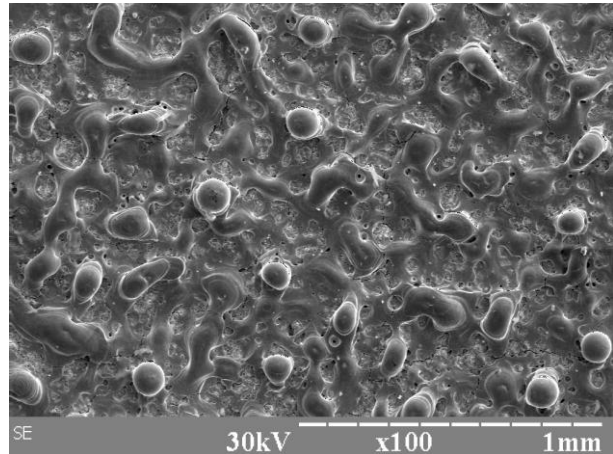


Fig. 4. SEM image of exposed surface

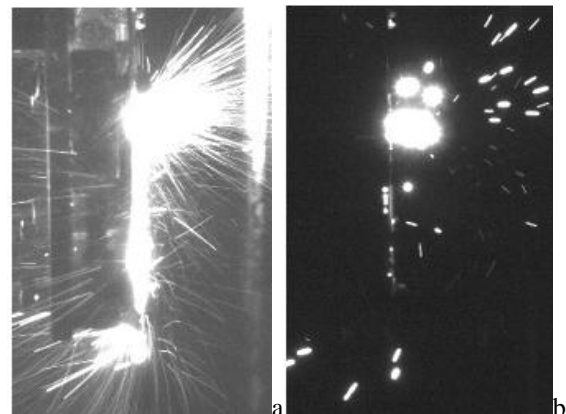


Fig. 5. High speed images of the tungsten coatings exposed to plasma of  $0.75 \text{ MJ/m}^2$ , as taken for  $t = 1.2 \text{ ms}$  and  $10.8 \text{ ms}$  after the beginning of plasma-surface interaction; the exposition time  $\tau_{\text{frame}} = 1.2 \text{ ms}$

After the hydrogen plasma loading, the surface stresses are annealed while being unchanged at the depth of the coating (see Fig. 2). The lattice parameter increases a little up to  $0.3160 \text{ nm}$  in the surface layer of  $0.2 \mu\text{m}$  (see Fig. 3). None of stress or lattice parameter variations were found at the depth of  $1 \mu\text{m}$ . However, closer to the surface, the lattice parameter increased due to vacancy annealing in the near surface layer.

Since the tensile stresses have occurred only within a thin surface layer, the compressive stresses might be developed at the depth of more than  $1 \mu\text{m}$  (Fig. 6). We suppose that these stresses are the result of twins' formation.

The plasma irradiation promoted increasing the coherence length from  $60$  to  $80 \text{ nm}$ . The average microstrains being initially about  $1.7 \times 10^{-3}$  have been

completely annealed as well as the initial dislocation density ( $9.2 \times 10 \text{ cm}^{-2}$ ) has lowered twice in the exposed surface layer. These facts indicate the hydrogen plasma irradiation main influence as the thermal loading on the surface of the coating.

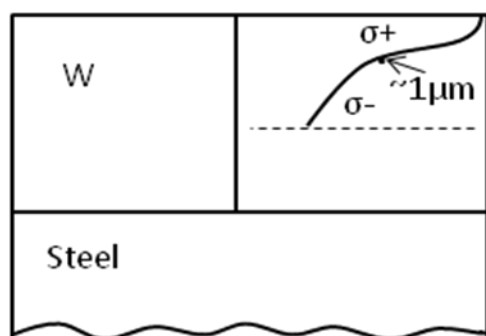


Fig. 6. A scheme of stress distribution over the coating tungsten depth

### CONCLUSIONS

As result of plasma irradiation, the texture with axis [100] normal to the surface appeared due to recrystallization of the affected material. Hydrogen plasma influence results also in decreasing the macro-stresses in the surface layer, increasing the coherence length from 60 nm in the initial state to 80 nm after the plasma irradiation. The initial average micro-strains about  $1.7 \times 10^{-3}$  were completely annealed, and the initial dislocation density ( $9.2 \times 10 \text{ cm}^{-2}$ ) has been lowered twice in the exposed surface layer. No structure and

stress variations were observed already at the depth near  $1 \mu\text{m}$  of the coating.

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### ЭВОЛЮЦИЯ СТРУКТУРЫ ВОЛЬФРАМОВЫХ ПОКРЫТИЙ, ОБЛУЧЁННЫХ ПОТОКАМИ ПЛАЗМЫ, ИМИТИРУЮЩИМИ УСЛОВИЯ ELM В ИТЭР

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Методами рентгеновской дифракции изучено изменение параметров структуры, субструктуры и напряжённого состояния на поверхности и по глубине покрытий вольфрам/сталь после облучения потоком водородной плазмы. Облучение плазмой приводит к рекристаллизационным процессам в тонком поверхностном слое покрытия: появлению текстуры [100] по нормали к поверхности, увеличению областей когерентного рассеяния от 60 нм в исходном состоянии до 80 нм после облучения, полному отжигу микронапряжений, а также уменьшению в два раза плотности дислокаций. Это подтверждает термический характер воздействия водородной плазмы. Уже на глубине около 1 мкм в покрытии никаких структурных изменений не выявлено.

### ЕВОЛЮЦІЯ СТРУКТУРИ ВОЛЬФРАМОВИХ ПОКРИТТІВ, ОПРОМІНЕНИХ ПОТОКАМИ ПЛАЗМИ, ЩО ІМІТУЮТЬ УМОВИ ELM В ІТЕР

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Методами рентгенівської дифракції вивчено змінення параметрів структури, субструктури та напруженого стану на поверхні та по глибині покриттів вольфрам/сталь після опромінення потоком водневої плазми. Опромінення плазмою призводить до рекристалізаційних процесів у тонкому поверхневому шарі покриття: виникненню текстури [100] по нормалі до поверхні, збільшенню областей когерентного розсіювання від 60 нм у початковому стані до 80 нм після опромінення, повному відпаду микронапружень, а також зменшенню у двічі густини дислокацій. Це підтверджує термічний характер впливу водневої плазми. Вже на глибині біля 1 мкм у покритті ніяких структурних змін не виявлено.