THERMAL GRAIN BOUNDARY GROOVES FORMATION IN TUNGSTEN UNDER RECRYSTALLIZATION

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Tungsten has been chosen as the main candidate for plasma facing components (PFCs) due to its superior properties under extreme operating conditions in future nuclear fusion reactors such as ITER and DEMO. One of the serious issues for PFCs is the high heat load during transient events and disruption in the reactor. Recrystallization and grain size growth in PFC materials are undesirable changes in the materials, since the microstructure developed after recrystallization exhibits a lower mechanical strength and an increased surface roughening. The current work was focused on careful investigation of the thermal grooving at grain boundaries (GB) in tungsten surface under recrystallization (W-rc). Topography of GB thermal grooves in W-rc is studied by atomic force microscopy (AFM). The peculiarities of its profile formation and the main factors which effect on the profile of GB grooving were determined. It is the purpose of this paper to point out these factors. The nature of GB grooving was determined.

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INTRODUCTION

Tungsten (W) is now considered as a perspective material for the elements which are plasma facing components (PFCs) in the International thermonuclear experimental reactor (ITER) [1]. Specifically for the first mirrors (FM) for plasma diagnostics. First mirror turned to plasma with its working surface is the responsible element of optical systems of optical and laser plasma diagnostics in ITER. FMs will be subjected to influence of all kind radiations emanating by burning plasma leading to degradation of their optical properties. Expected in ITER high heat load during plasma disruptions may result in distortion of ~ 100 μm W surface layer. In this case ~ 50 μm near-surface layer will be recrystallized. This will lead to embrittlement and cracking, which is dangerous for the tungsten structural elements. The main obstacle for structural applications of W-rc is its severe intrinsic GB brittleness. That is why among the candidate tungsten-based compounds for ITER which are now comprehensively tested in laboratories, significant attention is paid to recrystallized tungsten (W-rc). The knowledge of GB structure is important for understanding the mechanism of intergranular failure in W-rc. GB affect functional properties of tungsten. GB can not be identified on the as-received mirror polished FM surface even using an electron microscope. Recrystallization leads to thermal grooving [2], which are formed at the intersections of the grain boundary with the polished surface, and, thus, to surface damage and roughness formation, which will lead to the strongest impact on the mirror performance. Surface erosion by charge-exchange atoms can significantly change optical properties of mirrors and lead to a shutdown of the respective diagnostics. When the total area of the recrystallized W thermal grooves reaches 25% of the surface area and becomes comparable to the whole mirror surface area, it affects optical properties of tungsten mirrors [3], especially, polarization ones.

In this work, the results of a comprehensive study of W-rc surface are included. The problem of thermal grooves (TG) and triple junctions (TJ) as surface defects leading to specific surface roughness is discussed. Particular attention is paid to the conditions of TG grooves profile formation, their characteristics and nature. TG at GB in W-rc was studied by atomic force microscopy (AFM) technique. AFM combines the possibility to scan relatively large surface areas with the atomic resolution in the vertical direction, thus allowing determination of dihedral angle at the root of GB groove with a very high accuracy unattainable by other methods.

1. SAMPLES AND EXPERIMENTAL TECHNIQUES

Recrystallized tungsten was used in this work. The tungsten plates with purity of 99.99 wt.% and with 99.7% of theoretical density was prepared (A.L.M.T. Corp., Japan) by powder metallurgy and hot-rolled reduction. The plate was cut into specimens of 10×10×2 mm, doubled-sided mechanically and electrochemically polished to a high optical quality, and recrystallized at 2073 K for 1 h under hydrogen atmosphere.

Interferometric setup [4] and multipurpose optical setup [5] were used to study the surface relief. TG and their profiles were comprehensively examined with Scanning Electron Microscope (SEM; JSM 6390LV) equipped with an EBSD detector, and atomic-force microscope (AFM; C3M Solver P47-PRO). More experimental details can be found in [6–9].

2. SURFACE STRUCTURE, THERMAL GROOVES PROFILE AND GRAIN BOUNDARIES SHAPE (EXPERIMENTAL RESULTS)

The interference patterns of the W-rc sample surface show that the interference fringes inside the grains are straight and parallel that means that the grains surface is flat and smooth (not shown here). GB look like clear lines. In triple junction points one can see an equilibrium 120º contact angles between boundaries

which is specific for recrystallized materials. The typical grain size was in the range 20…250 μm. In previous publication [10] we drew attention to intricate profile of the GB in W-rc, specific for thermal grooves.

The AFM is a suitable technique to study the phenomenon of surface grooving at the GBs. It allows one to measure quantitatively the curvature of the groove profile. GB with various length were examined. Detailed examination reveals the GB grooves distorted by difference in the levels of two adjacent grains. It turned out, that equality in the levels of two adjacent grains [10] is just a special case of the grooves profile. As a rule, there are ridges of different levels and curvature, which is connected with the difference of surface energies of adjacent grains with different orientations and consistent with recent experimental results [11–13]. This conclusion is based on examination of very short GB (≤ 10 μm in our case) or of the part of GB near a triple junction. According to this conclusion, any thermal groove should have constant cross-profile along the GB dividing differently oriented grains. Difference between levels of adjacent grains may reach 1.1 μm (Fig. 1). The boundary, shown in Fig. 1, has a circle-arc curvature (it is marked with black dotted line). The boundary motion direction is indicated by black arrow, i. e., grain G1 was absorbing grain G2 under recrystallization. The number 1.1 μm indicates the height of the step between the levels of two adjacent grains.

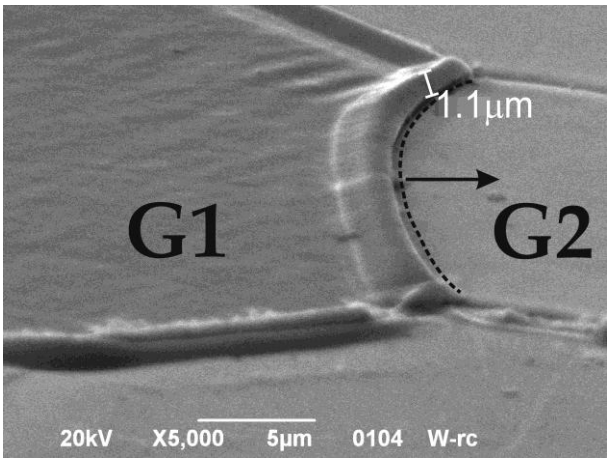

Fig. 1. SEM image of a grain boundary in the W-rc sample. Height difference on the GB is indicated by black line. Direction of the GB migration is indicated by black arrow

In this work many various grooves were investigated and analyzed. The groove shape was studied in details. A number of points were chosen along the GB on both sides of the GB groove between the minimum and maximum of the profile (for the left and right branches of the AFM profile). Fig. 2 illustrates the two typical AFM images of the GB grooves, together with the corresponding line topographic profiles. Fig. 2,a shows a 3D image for typical sufficiently short groove (the lateral size ≤ 10μm) near triple junction. The TG was profiled with 1 μm step along the boundary. Fig. 2,c shows the three TG profiles taken across the GB at different locations on the groove labeled 1, 2, and 3 in (a). All these three profiles exactly fit together qualitatively and quantitatively. At the same time, far from the groove root the level of the right grain in Fig. 2,a is significantly lower than the level of the left grain.

Recrystallization results in equilibration of the triple junction between the grain boundary and the free surface [14]. This equilibrium is set up almost instantaneously at high temperature and so the free surface, adjacent to the line where the GB emerges, becomes tightly curved as Fig. 2,b shows. From Fig. 2,c,d it can be seen a level difference between those of left and right grains.

For long grooves (the lateral size > 10 μm), TG profile is different at different locations along the groove. AFM image of such long GB (the lateral size 43 μm) is given in Fig. 2,b. This GB was profiled in detail (with 1 μm step along the boundary). Fig. 2,d shows the line scans of the AFM image in the direction perpendicular to the GB. AFM image of the area along the long TGB indicates that the level alignment between those of the left and right grains is the alternating quantity. Fig. 2,d shows three profiles measured in the vicinity of the GB inflection point, namely, two profiles were obtained at locations with curvature of opposite signs (profiles N 1 and 3), and the third one at the zero curvature location (profile N 2).

It has been found, that curved GBs are characterized by TG profile redistribution (see Fig. 2,d), profiles are vertically shifted to clarify. In the zero curvature location (inflection point) TG profile is symmetric: it can not be seen a level difference between those of left and right grains (see asymmetric profile N 2, Fig. 2,d). Before GB inflection point the level of the left grain is higher than the level of the right one (see profile N 1, Fig. 2,d), whereas after the inflection point the level of the left grain is lower than the level of the right grain (see profile N 3, Fig. 2,d).

AFM data for a great many of TG showed that some relative shift of the adjacent grains was observed for the majority of studied GBs, leading to the step between the levels of two adjacent grains. The possible reasons for the relative shift of adjacent W grains under recrystallization are the main goal of this work. For long boundaries this effect is variable and depends upon the boundary shape. Therefore, the boundaries form and its influence on TG profile formation have been studied in more detail. Under recrystallization grooves can interfere with the grain boundary advance on the surface. When this occurs, traces of old grooves ("ghost" traces) can be observed on the surface, showing the advance of GB while W grains are still growing. An example of this phenomenon is shown in Fig. 3, where the W grain is revealed with some "ghost" traces. It turned out, that on the surface one can see not only GB formed during recrystallization (see Fig. 3,a, black lines), but also "ghosts" (see Fig. 3,b, dotted lines), which are traces of boundaries of grains, which were absorbed during recrystallization, leaving a memory.

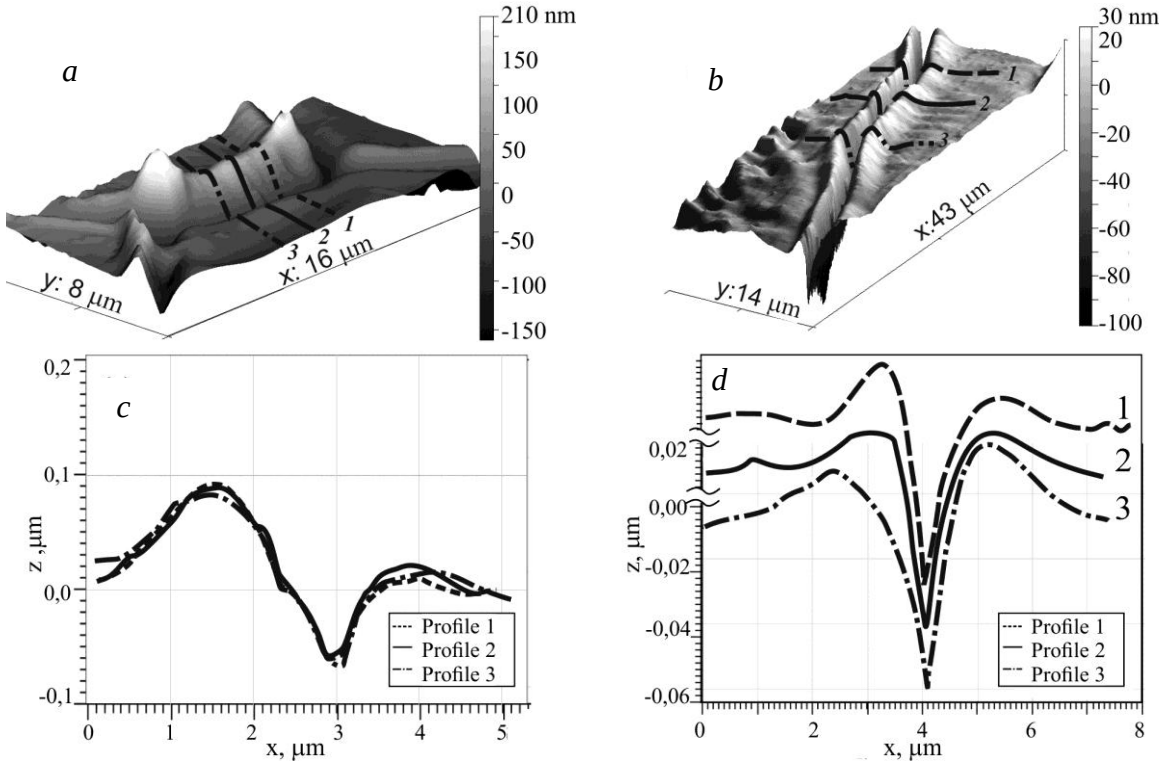

Fig. 2. Typical AFM 3D images of straight short (a) and curved long (b) GB and its linear AFM profiles (c and d, correspondingly) taken across the GB at different locations on the groove labeled 1, 2, 3 in a and b

GB "ghosts" formed by different mechanisms of GB motion during recrystallization are considered in more detail. Minimization of GB energy with the growth of a grain by reducing GB length is the main reason for the collective recrystallization. This process can be based on the two mechanisms: 1) migration of GB and 2) change in grains orientation [15].

In the first case, the migrating GB shifts towards the center of its curvature. Grains with concave boundaries absorb grains with convex ones. Since convex boundaries are more specific for small grains and concave ones for large grains, larger grains grow at the expense of the smaller grains. The light microscopy (LM) image of the studied area is shown in Fig. 3. Grains G1, G2, and G3 were adjacent to concave grain G4 (see dotted line in Fig. 3,b). Grains migrated towards the center of curvature (see white arrows in Fig. 3,b), moving dislocations in the same direction. When the dislocation density in the small grain G4 reaches a critical value it is energetically favorable to move abruptly boundaries of the grains G1, G2, G3 and to merge them into a triple junction. At the same time the grain G4 vanishes (see Fig. 3,c) and the total energy of the system decreases. G4 grain dislocations either annihilate or remain in GB and TJ. This process is illustrated schematically in Fig. 3,d.

In the case of the second recrystallization mechanism, boundaries vanish as a result of a grain rotation to merge with the lattice of the adjacent one. As a rule, such grains have low-angle boundaries [16]. Fig. 4 demonstrates traces of such kind of junction: initially there were two grains G1 and G2 (see Fig. 4,a). The boundary vanishes owing to the G2 reorientation, when it merges with G2. At this moment the recrystallization stops but the boundary traces remain (see indicated by white arrow in Fig. 4,b). The terminal points of the trace are former the two GB triple junctions (see TJ in Fig. 4,a). In this points real boundary remained curved, which is indicated by black arrows in Fig. 4,b. Also, in such points TG profile changes.

Fig. 5,a shows AFM image of a thermal groove and a "ghost" trace. Their profiles are presented in Fig. 5,b. Black line in Fig. 5,a shows the location at which the topographic trace in b was obtained. "Ghost" is a residual thermal groove, which does not separate two differently orientated grains now. Hence, its ridges (~ 20 nm height) are much lower, than the ridges of TG (~ 100 nm). Besides, the "ghost" groove is smoothed and less deep. Recrystallization is a thermally activated process, i. e., the system gets enough energy for boundary migration and grains merge (atomic diffusion). Hence, in the course of time, boundary "ghosts" vanish under the action of surface tension and the surface becomes smooth and flat. Not all boundaries are accompanied by "ghosts", the latter can be found only near recently formed boundaries.

The reason of a profile form variation near the intersection point of a real boundary and a "ghost" is clear from the boundary shown in Fig. 6. One can see a "ghost" trace on the right of the real boundary between grains G1 and G2. It means that earlier there was one more grain G3 not far from the groove root. Distinction between orientations of neighboring grains is one of the main characteristics which determine TG profile.

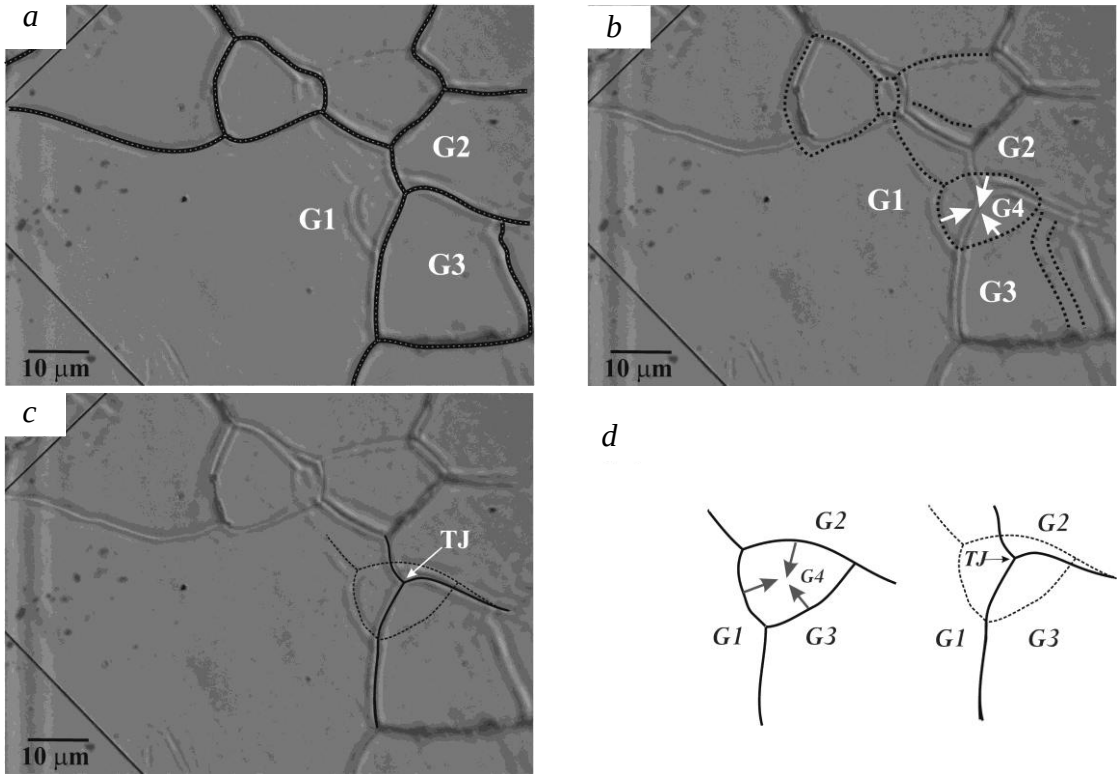

Fig. 3. Formation of "ghosts" as a result of GB migration (black lines mark the GB formed during recrystallization): a – LM image of the three grains G1, G2, and G3; b – "ghosts" – the traces of boundaries of grains, which disappeared during recrystallization (grain G4 boundaries are shown by dotted line, white arrows indicate the boundaries migration directions); c – a "ghost" formation as a result of a small grain G4 absorption when the GBs collapsed into the triple junction (dotted line marks the trace of the vanished boundary; dashed line – a real boundary); d – schematic illustration of "ghost" formation process

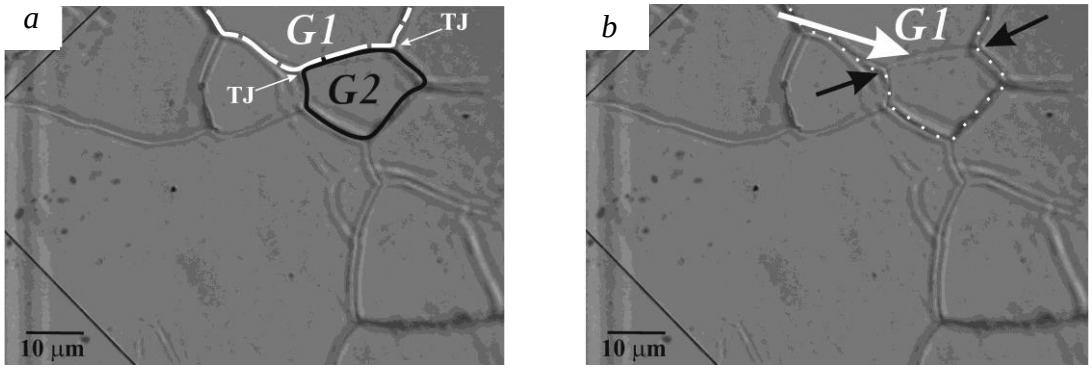

Fig. 4. Formation of "ghosts" as a result of GB vanishing when grains merge through rotation: a – white and black lines indicate boundaries of grains G1 and G2, correspondingly (TJ are triple junctions); b – white dotted line shows boundaries of the integrated grain G1, formed through grain merge (boundary curvature are indicated by black arrows; white arrow points out the "ghost" of the vanished boundary)

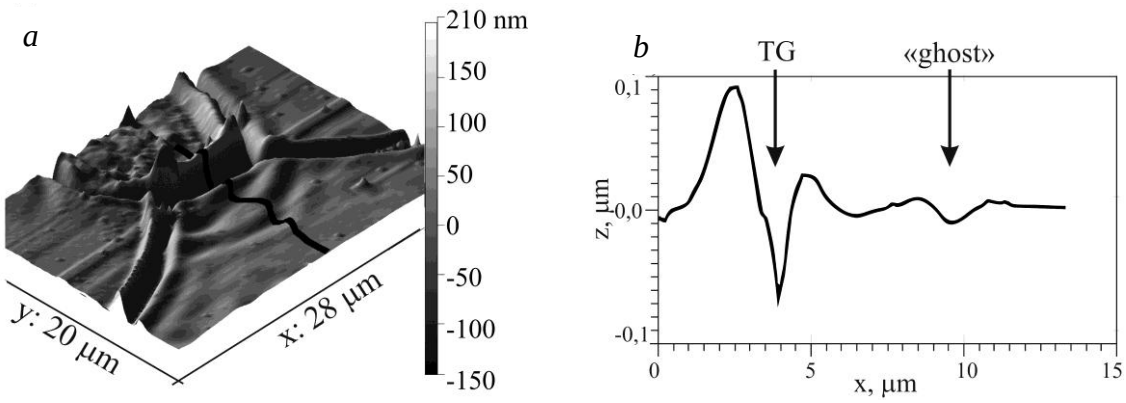

Fig. 5. AFM image of GBG with "ghost" trace (a); their profile taken across the black line in a (b)

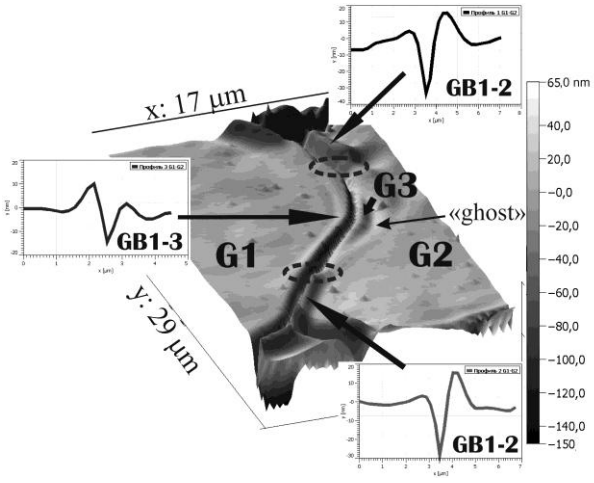

Fig. 6. AFM image of a real boundary between adjacent grains G1 and G2, and a "ghost" (a crescent-shaped indentation) to the right of the boundary. The inserts show TG profiles measured at the locations indicated by the corresponding arrows

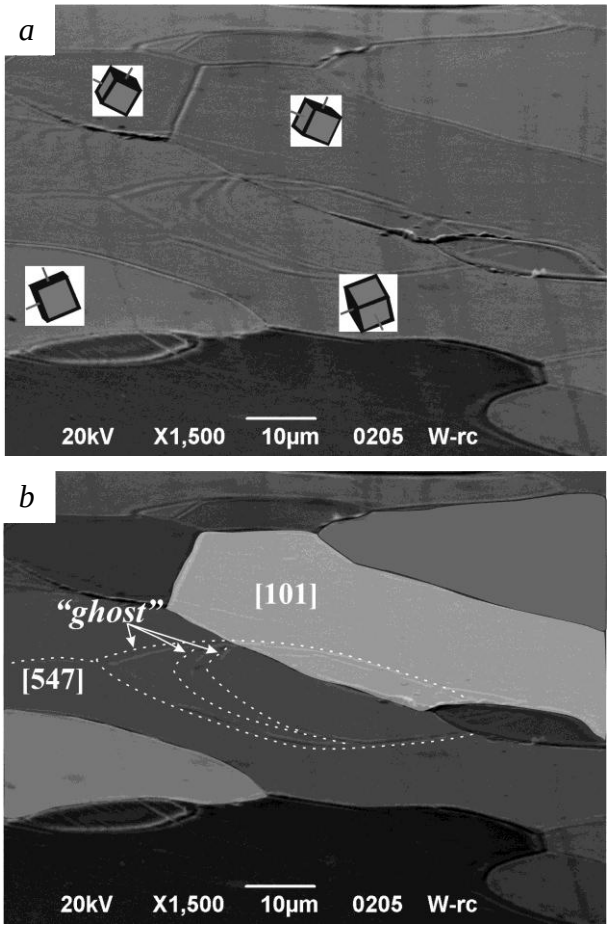

Fig. 7. SEM (with EBSD) image of the W-rc sample surface. Grains orientation, indicated by the cube inserts, was determined by EBSD (a); color of separate grains is uniform regardless of the "ghosts" (dotted line) (b)

So, it is natural to assume that in a polycrystal boundaries between equal pairs of grains should have at least similar TG profiles. If all the characteristics, which determine TG profile, coincide, the profiles should be equal. In terminal points (they look like dotted line in Fig. 6) the boundary separates grains G1 and G2. GB1-2 profiles near these points are equal: the level of the left grain is lower than the level of the right grain (see inserts in Fig. 6). In the region where G1 formerly bordered on grain G3 (see GB1-3 in Fig. 6) the TG profile has changes: the level of the right grain is lower than the level of the left grain. Despite G3 does not exist already, the TG "remembers" the previous profile.

According to EBSD analysis, grain areas adjacent to "ghosts" have the same crystallographic orientation as the whole grain. SEM image of the studied area with schematic representation of grains orientation by cubic inserts is shown in Fig. 7,a. Fig. 7,b shows the same area with color coded orientation map and their Miller indices indicated. Grey grain [547] in the center of the image has "ghosts", but its color is uniform.

Pinning of migrating large GB (longer than the smallest grain) by defects during recrystallization, leads to bends formation as well. Keeping linear shape of a large boundary is improbable, since always there are defects over long distances in real crystals. So, any real long boundary is always curvy.

CONCLUSIONS

Results of detailed experimental studies of the effect of recrystallization on tungsten surface structure formation are presented. The topography of GB grooves, GB in W-rc and the "ghost" traces were studied using AFM. It was found that W-rc surface consists of grains separated by complicated GB with TG formed during recrystallization, which leads to formation of a complex relief of the surface. A more detailed examination by AFM revealed "ghost" traces, i. e., locations of grains absorbed during recrystallization.

Investigations of TG were started in W.W. Mullins theoretical study [17]. Mullins developed a theory of GB in terms of the mechanisms of surface diffusion, volume diffusion, and evaporation-condensation. AFM have shown that the surface morphology in the region of GBG is much more complex, than is implied by the classical profile analyzed by W.W. Mullins. According to Mullins, TG is a result of free surface and GB surface tension balance. Mullins theory, described the mechanism of GB grooving, gave a quantitative treatment of the problem for sufficiently small grooves (in typical metals the lateral size of the groove should be less than ~ 10 μm [18]). Further experimental studies revealed a typical Mullins-type profile distorted only by difference in the levels of two adjacent grains. This fact was explained by surface tension distinction for different grains. Since the surface tension of a grain is a constant, the TG profile should be the same along the GB.

Currently, from references we know either modeling results or TG profiles near triple junction only. In this study it shown experimentally for the first time that to understand the nature of the ridges (i. e. driving forces during recrystallization) the whole long boundary should be considered. A great number of TG was examined and most specific are presented.

Our experiments revealed that real TG profile is asymmetric and it is constant in the case of very short

boundaries only. For most long boundaries the profile is asymmetric and variable along the boundary.

Identification of the nature of variable TG profile was the purpose of the present study. It is shown for the first time that peculiarities of GBs movement during recrystallization make a decisive contribution to TG profile formation. Namely, in real crystals various defects act as pinning centers for the boundaries. In addition, traces of the former boundaries (so-called "ghosts") remain on the surface. It is shown that these factors make the boundaries curve. If GB grooving is accompanied by an old grain – "ghosts", the relationship between the groove width and depth, derived by Mullins, is no long valid.

The main result of this study is that profile of a TG, formed on the surface of tungsten during recrystallization, is variable along GB. It is defined by the GB history and the crystal peculiarities, such as "ghosts" and pinning centers. It is shown for the first time that TG profile asymmetry depends on the grain boundary shape during recrystallization.

So, we can make the following conclusion, based on this study. Profile of a thermal groove, formed during recrystallization, is different at different cross-section locations along the GB, and it is defined by boundary shape in real crystal and depends upon the boundary history. Any boundary curvature affects the thermal groove profile (ridges height, asymmetry, etc.). Thus, surface energy, which depends on the grain orientation, is not the decisive factor of TG profile formation.

The role of pinning is rather clear, whereas the one of "ghosts" requires further comments.

Physical reasons of "ghosts" formation can be explained as follows. After jerky GB migration atoms should move from high concentration region ("former" ridges) to the low concentration one ("former" groove) due to difference of atoms concentration in the system; i. e. surface self-diffusion should occur. This process takes some time, but recrystallization is terminated and the atoms do not have enough time to redistribute according to new surface and GB energies (tensions). As a result the TG "remembers" information about "former" profile, since when recrystallization is terminated atoms lack energy for further diffusion.

It is shown that GB and triple junctions, formed as a result of recrystallization, are the most important elements of W-rc structure. While GB has been studied thoroughly over a long period of time, TJ became the subject of study as a special type of a crystal defect not long ago. Specific structure, thermodynamic and kinetic properties are inherent to such objects and in many cases they determine processes in polycrystalline materials.

Results of the present study of individual TJs motion showed for the first time, how much they can affect the grain growth process in polycrystalline W. GB (thermal grooves) and TJ are surface defects which form the surface roughness substantially. Roughness necessarily affects the optical parameters of the FM, since reflectivity is their main characteristics.

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ФОРМИРОВАНИЕ ТЕРМИЧЕСКИХ КАНАВОК ТРАВЛЕНИЯ НА ГРАНИЦАХ ЗЕРЕН ВОЛЬФРАМА ПРИ РЕКРИСТАЛЛИЗАЦИИ

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Благодаря своим уникальным свойствам вольфрам является основным кандидатом для обращенных к плазме конструкционных элементов таких перспективных реакторов, как ITER и DEMO. Одной из серьезных проблем для таких элементов является высокая термическая нагрузка при переходных процессах и срывах плазмы в реакторе. Рекристаллизация и увеличение размера зерна в материалах, обращенных к плазме элементов, являются нежелательными, поскольку микроструктура, формирующаяся при рекристаллизации, дает более низкие механические свойства и увеличенную шероховатость. Настоящая работа посвящена исследованию формирования термических канавок на границах зерен в вольфраме при рекристаллизации (W-rc). Топография канавок термического травления (КТТ) в W-rc изучена с помощью атомной силовой микроскопии. Определены особенности формирования КТТ и основные факторы, влияющие на их профиль. Целью данной работы было исследование этих факторов. Установлена природа термических канавок травления.

ФОРМУВАННЯ ТЕРМІЧНИХ КАНАВОК ТРАВЛЕННЯ НА ГРАНИЦЯХ ЗЕРЕН ВОЛЬФРАМУ ПРИ РЕКРИСТАЛІЗАЦІЇ

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Завдяки своїм унікальним властивостям вольфрам є основним кандидатом для конструкційних елементів, які повернені до плазми, у таких перспективних реакторах, як ITER та DEMO. Однією з важливих проблем для таких елементів є високе термічне навантаження при перехідних процесах та зривах плазми в реакторі. Рекристалізація та зростання розміру зерна в матеріалах елементів, які повернені до плазми, є небажаними, оскільки мікроструктура, що формується при рекристалізації, дає більш низькі механічні властивості та підвищену шорсткість. Ця робота присвячена дослідженню формування термічних канавок на границях зерен у вольфрамі при рекристалізації (W-rc). Топографія канавок термічного травлення (КТТ) у W-rc вивчена за допомогою атомної силової мікроскопії. Визначені особливості формування КТТ та основні фактори, що впливають на їх профіль. Метою даної роботи було вияснення цих факторів. З'ясована природа термічних канавок травлення.