Materials Research Express

PAPER

Texture evolution of tungsten materials during recrystallization

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Keywords: pure tungsten, lanthanum oxide doped tungsten, rolling texture, recrystallization texture

Abstract
Some functional and structural performance of tungsten (W) are related to its texture characteristics. Usually, W serves in high temperature and may undergo recrystallization. Thus it’s necessary to evaluate the recrystallization texture of W. In our previous studies, pure W (PW) and W-1.0wt%La2O3 (WL10) were deformed via unidirectional rolling (UNR), cross rolling (CRR) and clock rolling (CLR) with 80% reduction. In the present paper, PW-UNR, PW-CRR, PW-CLR and WL10-UNR were subjected to annealing at 2073 K for 2 h to achieve recrystallization and figure out the evolution mechanism of recrystallization texture in W materials. Besides, the effect of La2O3 on recrystallization texture of W was discussed. The results indicated that the fiber textures in rolling state were transformed into isolated textures after recrystallization. {001}〈uvw〉 isolated texture formed in the recrystallized W may be mainly resulted from the texture inheritance. {113}〈361〉 isolated texture formed in the recrystallized WL10-UNR may be attributed to the oriented growth of {113}〈361〉 grains with high grain boundary mobility facilitated by La2O3. Generally, isolated textures near θ-fiber were strengthened while γ-fiber was weakened for W during recrystallization, which is an effective method to achieve W with more {001} and less {111} textures.

1. Introduction
Tungsten (W) and its alloys have been widely used as the functional and structural materials in nuclear, electronic, aerospace, military, manufacturing, coating and energy industries owing to their brilliant characteristics such as high melting point, high thermal conductivity, high chemical stability, low thermal expansion and excellent high temperature mechanical properties [1–3]. Usually, W serves in high temperature and may undergo recrystallization. For example, W is considered as the plasma facing material (PFM) in future fusion reactor and will be subjected to intensive heat loads, which will result in the rapid temperature increase of W and further lead to recrystallization and grain growth [4–7]. Thus the functional and structural performance of W after recrystallization should be concerned.

It has been indicated that the functional properties and structure performance of W in terms of particle irradiation resistance [8–17], surface morphology after irradiation [18–21], thermal conductivity [22] and fracture toughness [23] were related to its crystal orientation. Specifically, W grains with low crystallographic index displayed better irradiation resistance under deuterium plasma [8], helium neutral beam [9], helium/hydrogen ions [10] and Ga+ ion [11]. Surface morphology after irradiation was correlated with the normal direction of grain [19]. The close-packed {111} direction exhibited the largest thermal conductivity [22]. {110} crack systems exhibited higher fracture toughness at room temperature compared to the {100} ones [23]. Thus it is very necessary and significant to investigate the texture characteristics of W after recrystallization.

Recrystallization texture of W has been characterized via Electron Back-Scattered Diffraction (EBSD) technique [24–28]. In [24], γ and α fiber rolling textures were replaced by random texture with a slight cube component in recrystallized W due to the oriented nucleation of cube nuclei. In [25], deformation textures of {001}〈110〉 and {111}〈110〉 were remained after normal recrystallization. Meanwhile, {001}〈010〉 and {111}
(112) textures were strengthened after recrystallization due to the subgrain coarsening mechanism. In [26], (100) (011) rolling texture became more pronounced as the annealing temperature increased. In [27], the rolled W foil showed a pronounced (100) (011) texture while the annealed one displayed a texture varying between (100) (011) and (112) (011). In [28], surface recrystallization was achieved on the severely deformed W and cold rolled W via transient high heat flux of laser beam. The severely deformed W exhibited random orientation while the cold rolled one showed the {hkl} (001) orientation. After recrystallization, grains remained random orientation in the severely deformed W. In the case of the cold rolled one, (001) direction became preferential again after the nucleation phase for higher power loads and number of pulses, which was attributed to the selected growth of grains preferably oriented with respect to the matrix. Obviously, recrystallization texture characteristics of W measured via EBSD varied significantly.

In fact, EBSD is suitable for measuring the crystal orientation for single or several grains. And the statistical texture characteristics for lots of grains should be determined by x-ray Diffraction (XRD) instead. In other words, EBSD and XRD are more suitable to determine the micro-texture and macro-texture, respectively [29]. Recrystallization texture of W foil was measured by XRD in [30] and the results indicated the {100} (011)±12° texture was developed after annealing at temperature above 2073 K Y B Park et al attributed the formation of {100} (011)±12° recrystallization texture to growth selection [31, 32]. But the evolution of recrystallization texture in rolled W sheets has never been determined via XRD. In our previous studies, pure W (PW) and W-1.0wt%La2O3 (WL10) with 80% total reduction were plastic deformed via unidirectional rolling (UNR), cross rolling (CRR) and clock rolling (CLR) [33, 34]. Thus in the present study, PW-UNR, PW-CRR, PW-CLR and WL10-UNR will be subjected to annealing at 2073 K for 2 h to achieve recrystallization and figure out the evolution mechanism of recrystallization texture in W materials via XRD technique. Furthermore, the effect of second phase of La2O3 on the recrystallization texture of W will be discussed too.

2. Materials and characterization

The starting powders of PW and WL10 with grain size of about 3 μm were commercial. The chemical compositions of both powders have been shown in [33]. Firstly, the W powders were subjected to powder metallurgy to obtain the sintered billets. Cold isostatic pressing was performed at 200 MPa for 2 min and medium-frequency induction sintering was carried out at 2373 K for 2 h in hydrogen atmosphere. Subsequently, PW and WL10 billets were rolled via an industrial rolling mill. For PW, the billets were rolled by UNR, CRR and CLR, respectively. For WL10, the billet was rolled only by UNR. The schematic diagrams of UNR, CRR and CLR have been illustrated in [34]. For the UNR, rolling direction was always the same. For the CRR, rolling direction was clockwise rotated 90° and 270° alternately compared to the previous pass. For the CLR, rolling direction was clockwise rotated 135° compared to the previous pass. The initial thickness of the sintered billets was about 28 mm and the final thickness of the rolled plates was about 5.6 mm. The total thickness reduction of about 80% was obtained through 8 passes. Then these deformed sheets were subjected to the stress relieving at 1373 K in hydrogen atmosphere for 2 h. Finally, the PW and WL10 samples were annealed at 2073 K for 2 h in argon atmosphere using a vacuum hot-press furnace (ZT-60-23Y3) to achieve recrystallization. Recrystallized PW-UNR, PW-CRR, PW-CLR and WL10-UNR were named as PW-UNR-REC, PW-CRR-REC, PW-CLR-REC and WL10-UNR-REC, respectively. Samples for texture measurement and microstructure examination were sectioned along the final rolling direction and the texture measurement specimens were cut from the RD-TD surface and the microstructure examination specimens were cut from the RD-ND surface, respectively. RD, TD and ND are short for rolling direction, transverse direction and normal direction, respectively.

Microstructure was examined by scanning electron microscopy (SEM) and the grain boundaries were etched via standard Murakami’s solution (1:1:10 for KOH:K3Fe(CN)6:H2O). Texture was measured by XRD. Orientation distribution functions (ODFs) were used to describe the texture characteristics of W before and after recrystallization. Before texture measurement, specimen surface (15 × 15 mm2) was mechanically polished. Then three incomplete pole figures (PFs) of {110}, {200}, {211} in the center layer (RD-TD plane) of the samples were measured. This measurements were conducted with the pole distance angle ranging from 20° to 90° in the back reflection mode using Cu Kα1 radiation. During the measurements, specimens were oscillated along TD to cover larger specimen area. Finally, ODFs were computed based on the three PFs.

3. Results

θ-fiber, γ-fiber and {113}〈361〉 textures shown in figure 1 were used to evaluate the texture evolution of PW-UNR, PW-CRR, PW-CLR and WL10-UNR during recrystallization. {001}〈110〉, {001}〈310〉, {001}〈100〉 are the main textures along θ-fiber and {111}〈110〉, {111}〈112〉 are the main textures along γ-fiber.
ODFs of the unidirectional rolled PW before and after annealing are shown in figure 2. For the rolled sample, \( \theta \)-fiber and \( \gamma \)-fiber textures were formed simultaneously. Furthermore, the intensity of \( \theta \)-fiber texture was higher than \( \gamma \)-fiber texture. Besides, \{001\}\langle110\rangle component displayed higher intensity of about 11.5 compared to 7.7 for the \{001\}\langle100\rangle component. For the annealed one, two kinds of isolated textures near \( \theta \)-fiber formed. One can be approximately regarded as \{001\}\langle110\rangle and the other one can be roughly considered as \{001\}\langle100\rangle. Furthermore, the intensity values of \{001\}\langle110\rangle and \{001\}\langle100\rangle were about 19.6 and 17.5, respectively, which were higher than those in the rolled sample. In brief, PW-UNR exhibited \( \theta \)-fiber and \( \gamma \)-fiber textures while PW-UNR-REC mainly presented isolated textures near \( \theta \)-fiber including \{001\}\langle110\rangle and \{001\}\langle100\rangle, which were strengthened significantly after annealing. Moreover, except for the isolated textures, PW-UNR-REC exhibited random texture characteristics.

Figure 3 shows the ODFs of the cross rolled PW before and after annealing. For the rolled sample, \{001\}\langle110\rangle texture and \( \gamma \)-fiber texture were formed simultaneously. The dominant component in \( \gamma \)-fiber was \{111\}\langle110\rangle. The intensity values of the \{001\}\langle110\rangle and \{111\}\langle110\rangle textures were about 11.5 and 10.0, respectively. For the annealed sample, three kinds of isolated textures formed near \( \theta \)-fiber, i.e., \{001\}\langle110\rangle, \{001\}\langle310\rangle, \{001\}\langle100\rangle with the intensity value of 16.7, 10.0, 12.9, respectively. Besides, some weak isolated textures with
intensity of 4.9-8.5 formed along γ-fiber. The comparison between PW-CRR and PW-CRR-REC demonstrated that the textures near θ-fiber including {001}⟨110⟩, {001}⟨310⟩ and {001}⟨100⟩ were strengthened while textures along γ-fiber were weakened. Furthermore, except for the isolated textures, PW-CRR-REC exhibited random texture characteristics, which was similar to the results in PW-UNR-REC.

Figure 4 shows the ODFs of the clock rolled PW before and after annealing. For the rolled sample, {001}⟨110⟩ texture and γ-fiber texture formed simultaneously. The dominant component in γ-fiber was {111}⟨112⟩, {001}⟨110⟩ and {111}⟨112⟩ textures exhibited close intensity value of about 7.3. For the annealed sample, three kinds of isolated textures formed near θ-fiber including {001}⟨110⟩, {001}⟨310⟩, {001}⟨100⟩ with the intensity of 12.2, 11.2, 11.8, respectively. Besides, isolated texture of {111}⟨112⟩ with the intensity of 13.1 formed along γ-fiber. The comparison between the PW-CLR and PW-CLR-REC demonstrated that the textures near θ-fiber including {001}⟨110⟩, {001}⟨310⟩ and {001}⟨100⟩ were strengthened while textures along γ-fiber were weakened except for {111}⟨112⟩. Moreover, except for the isolated textures, PW-CLR-REC exhibited random texture characteristics, which was similar to the results in unidirectional and cross rolled PW.

Figure 5 shows the ODFs of the unidirectional rolled WL10 before and after annealing. For the rolled sample, θ-fiber and weak {112}⟨110⟩ textures were formed simultaneously. The dominant component in θ-fiber was {001}⟨110⟩, which displayed the intensity of about 13.8. For the annealed sample, three kinds of isolated textures formed. Textures near θ-fiber can be approximately regarded as {001}⟨100⟩ and {001}⟨310⟩ with about 5° offset from Φ. The third one exhibited Euler angles of ϕ₁ = 20°, ϕ₂ = 45° and Φ = 25°, which correspond to the Miller index of {113}⟨361⟩. Intensity value of {001}⟨100⟩, {001}⟨310⟩ and {113}⟨361⟩ texture was about 15.2, 12.2 and 20.1, respectively.

To figure out the texture evolution of θ-fiber and γ-fiber during annealing quantitatively, orientation line analysis before and after annealing was summarized and presented in figures 6–9. Considering that some...
isolated textures formed near θ-fiber in the annealed samples, several orientation lines along θ-fiber with 5°–20° offset from Φ were also analyzed. In figures 6(a), (b), PW-UNR-REC exhibited higher intensity in \{001\}(110), \{001\}(100) textures while lower intensity along γ-fiber compared to PW-UNR. In figures 7(a), (b), PW-CRR-REC showed higher intensity in \{001\}(110), \{001\}(310), \{001\}(100) textures while lower intensity along γ-fiber except for the \{111\}(143) component compared to PW-CRR. In figures 8(a), (b), PW-CLR-REC displayed

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**Figure 5.** ODFs (ϕ₂ = 45°) of the WL10-UNR (a) and WL10-UNR-REC (b).

**Figure 6.** Orientation line analysis of the unidirectional rolled PW before and after annealing: θ-fiber texture (a), γ-fiber texture (b).

**Figure 7.** Orientation line analysis of the cross rolled PW before and after annealing: θ-fiber texture (a), γ-fiber texture (b).
higher intensity in \{001\}\{110\}, \{001\}\{310\}, \{001\}\{100\} textures while lower intensity along γ-fiber except for the \{111\}\{112\} component compared to PW-CLR. In figures 9(a), (b), WL10-UNR-REC exhibited higher intensity in \{001\}\{310\}, \{001\}\{100\} textures while lower intensity along γ-fiber except for the \{111\}\{110\} component compared to PW-CLR. Generally, \{001\}\{110\}, \{001\}\{310\} and \{001\}\{100\} textures along θ-fiber were strengthened while texture along γ-fiber was weakened during annealing except for the \{111\}\{112\} component in PW-CLR-REC. Thus the formation of recrystallization random texture and recrystallization isolated texture will be discussed in the following section.

4. Discussion

4.1. Recrystallization random texture
The evolution of recrystallization texture in W can be explained via the model proposed by Park et al [31]. This model is composed of two principles: (i) The maximum principal stress direction in the deformed grain becomes the minimum Young’s modulus direction in the recrystallized grain; (ii) \{110\} plane paralleling to the maximum principal stress direction (hence, the minimum Young’s modulus direction) is taken for variant selection. The driving force of recrystallization can be expressed by the following formula (1):
where subscripts d and r denote the deformed and recrystallized states, respectively. \(\sigma\) is the acting residual stress. \(E\) is the Young’s modulus. \(\varepsilon\) is the compensating strain. \(\gamma_{ds}\) is the interfacial free energy per unit area. \(\kappa\) is a shape factor. \(D\) is the grain size. On the one hand, the energy release during expansion of the recrystallization front is highest for the maximum stress in the deformed state and minimum Young’s modulus in the recrystallization state, i.e., the release of stored energy is greatest if the direction of minimum Young’s modulus of the recrystallized grain lines up with the maximum principal stress of the deformed grain. On the other hand, the release of stored energy is less if the direction of minimum Young’s modulus is not in the same direction as the maximum principal stress of the deformed grain. On the other hand, initial deformed grains with high intensity of the survived texture were detected in PW-UNR.

4.2. Recrystallization isolated texture

Table 1 summarizes the main isolated texture components in the recrystallized PW and WL10. \{001\} (uvw) and \{111\} (112), \{113\} (361) were the main isolated textures. \{001\} (110), \{001\} (310), \{001\} (100) were the main isolated texture along \(\theta\)-fiber.

\[ P = \frac{\sigma^2}{2 E_d} - \frac{E \varepsilon^2}{2} - \frac{\gamma_{ds} \kappa}{D} \]  

(1)

\(D\) is the interfacial free energy per unit area.

\{001\} (110) isolated texture formed in PW-UNR-REC, PW-CRR-REC and PW-CLR-REC can be attributed to the following reasons: \{001\} (110) texture with high intensity was generated in PW-UNR, PW-CRR and PW-CLR samples as shown in figures 2(a)–4(a) due to the activation of \{112\} (111) slip system [29]. Usually, \{001\} (110) texture possesses low deformation stored energy as shown in [24] thereby would be consumed naturally during annealing.

\{001\} (100) isolated texture formed in PW-UNR-REC, PW-CRR-REC, PW-CLR-REC and WL10-UNR-REC may be resulted from the following reasons: on the one hand, recrystallization grains with \{001\} (100) orientation would be nucleated in the deformed grains with \{111\} (112) and \{001\} (100) orientations [35, 36]. On the other hand, initial deformed grains with \{001\} (100) orientation is quasi-stable and can be partially preserved during recrystallization due to the texture inheritance [37]. In fact, \{001\} (100) and \{111\} (112) textures were detected in PW-UNR. \{111\} (112) texture was formed in PW-CRR and PW-CLR. \{001\} (100) texture was observed in WL10-UNR. Therefore, high intensity of \{001\} (100) in the recrystallized PW and WL10 may be attributed to these initial rolling textures of \{111\} (112) and \{001\} (100).

\{001\} (310) texture exhibited lower intensity compared to the other isolated textures as shown in table 1. Furthermore, \{001\} (310) is the transitional texture between \{001\} (110) and \{001\} (100) along \(\theta\)-fiber. Thus \{001\} (310) may be transformed from \{001\} (110) and \{001\} (100) textures, which will be investigated via EBSD in the future.

\{111\} (112) texture disappeared almost in the recrystallized W except for in PW-CLR-REC. It has been proved that \{111\} (uvw) displays more deformation stored energy compared to \{001\} (uvw) [24, 31], and the texture with more stored energy will be consumed faster selectively. Thus the lower intensity in \{111\} (110) and \{111\} (112) textures would be attributed to the preferential growth of the recrystallized components into the rolling \(\gamma\)-fiber. In the case of the PW-CLR-REC, high intensity in \{111\} (112) texture may be resulted from the more initial texture of \{111\} (112) in PW-CLR. Although \{111\} (112) texture exhibits more deformation stored energy than the other ones, incomplete consumption may lead to the high intensity of the survived \{111\} (112) texture in PW-CLR-REC.

\{113\} (361) isolated texture only formed in the WL10-UNR-REC. Previous literatures indicated that the recrystallization texture of \{113\} (361) can be resulted from the deformation texture of \{001\} (110) [38] and \{112\} (110) [39]. Considering the fact that \{001\} (110) was detected in all the rolled PW, WL10 samples and weak \{112\} (110) formed in WL10-UNR, it can be deduced that the recrystallization texture of \{113\} (361) in WL10-UNR-REC should not be resulted from \{001\} (110) and \{112\} (110) completely. Sanjari et al [40] investigated the evolution of texture and microstructure in the rolled Si-containing steel during annealing via EBSD, and the results indicated that the deformation \{001\} (130) grains disappeared and recrystallization \{113\}
(uvw) grains formed gradually. Analysis of the misorientations between the deformed (001)〈130〉 grains and the neighboring recrystallized grains indicated that the preferred growth of the (113)〈uvw〉 grains might be due to the high grain boundary mobility of these grains with respect to the deformed matrix. Figure 10 shows the SEM micrographs of PW-UNR-REC and WL10-UNR-REC. Grain size of WL10-UNR-REC was smaller than that of PW-UNR-REC due to the grain boundary movement inhibition induced by La2O3 particles. Thus (113)〈361〉 grains with high grain boundary mobility would be developed preferentially in WL10-UNR-REC due to the grain boundary movement inhibition from La2O3 particles. Increasing the annealing temperature or annealing time may lead to sufficient grain boundary movement and thereby result in the reduction of (113)〈361〉 texture and increase of (001)〈uvw〉 texture, which will be examined in the future. Briefly, (113)〈361〉 isolated texture in WL10-UNR-REC may be resulted from the orientated growth induced by La2O3 particles as well as the transformation from the initial (001)〈110〉 and (112)〈110〉 components.

Besides, the volume fraction of (100), (111) and (110) textures in rolled and recrystallized PW and WL10 is shown in figure 11. Obviously, all the samples displayed the least (110) texture compared to (100) and (111) textures. Thus the comparison between (100) texture and (111) texture was mainly conducted in the later. PW-UNR-REC exhibited more (100) texture and close (111) texture compared to PW-UNR. PW-CRR-REC showed less (111) texture and close (100) texture compared to PW-CRR. PW-CLR-REC displayed more (100) texture and less (111) texture compared to PW-CLR. Thus it can be summarized that the recrystallized PW

Figure 10. SEM micrographs of PW-UNR-REC (a) and WL10-UNR-REC (b).

Figure 11. Volume fraction of (100), (111), (110) textures in rolled and recrystallized PW and WL10.
showed more $\{100\}$ texture or less $\{111\}$ texture. By contrast, WL10-UNR-REC exhibited less $\{100\}$ and $\{111\}$ textures simultaneously compared to WL10-UNR, which can be resulted from the formation of $\{113\}$ and $\{361\}$. Higher annealing temperature and longer annealing time may weaken the $\{001\}$ texture or increase the $\{111\}$ texture. Thus recrystallization is an effective method to increase $\{001\}$ texture and decrease $\{111\}$ texture in W.

5. Conclusions

In this study, PW-UNR, PW-CRR, PW-CLR and WL10-UNR with 80% deformation degree were subjected to annealing at 2073 K for 2 h to achieve recrystallization. Then the texture characteristics in the rolled and recrystallized W samples were measured to figure out the texture evolution during recrystallization. Besides, the effect of La$_2$O$_3$ on recrystallization texture of W was also evaluated. The conclusions drawn from the results can be summarized as follows:

1. Rolling fiber textures were transformed into isolated components in W during recrystallization. Except for the isolated textures, recrystallized W exhibited almost random texture due to the elastically isotropic.
2. Isolated textures near $\theta$-fiber were strengthened while $\gamma$-fiber was weakened for W during annealing.
3. $\{001\}$ texture formed in recrystallized W may be resulted from the texture inheritance.
4. $\{113\}$ and $\{361\}$ isolated texture formed in WL10-UNR-REC may be attributed to the orientated growth facilitated by La$_2$O$_3$ particles.
5. Recrystallization is an effective method to increase $\{001\}$ texture and decrease $\{111\}$ texture in W.

Acknowledgments

This work was supported by China Postdoctoral Science Foundation [2018M643167]; ITER-National Magnetic Confinement Fusion Program [2014GB123000] and National Natural Science Foundation of China [11905137, [51801171], [51601097].

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