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Published in:
IOP Conference Series: Materials Science and Engineering

Link to article, DOI:
10.1088/1757-899X/1121/1/012018

Publication date:
2021

Document Version
Publisher's PDF, also known as Version of record

Link back to DTU Orbit

Citation (APA):
Tarras Madsen, D., Ciucani, U. M., Hoffmann, A., & Pantleon, W. (2021). Strong rotated cube textures in thin cold-rolled potassium-doped tungsten sheets during annealing up to 1300 °C. IOP Conference Series: Materials Science and Engineering, 1121(1), [012018]. https://doi.org/10.1088/1757-899X/1121/1/012018

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To cite this article: D Tarras Madsen et al 2021 IOP Conf. Ser.: Mater. Sci. Eng. 1121 012018

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Strong rotated cube textures in thin cold-rolled potassium-doped tungsten sheets during annealing up to 1300 °C

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Abstract. Due to the high operation temperatures, microstructural changes will occur in tungsten when used as plasma-facing material in future fusion reactors. In drawn tungsten wires, potassium doping has proven to prevent such undesired changes effectively. This strategy has been adapted to thin cold-rolled tungsten sheets containing 80 ppm potassium. Their texture evolution during annealing is investigated using electron backscatter diffraction. In the as-rolled condition, a strong cube texture is present with a volume fraction of 67.0 % deviating maximal 15° from the rotated cube orientation. This texture intensifies strongly during isochronal annealing for 2 h at temperatures between 1000 °C and 1300 °C; the higher the temperature, the higher the rotated cube fraction. The volume fraction of 94.8 % obtained after 2 h at 1300 °C does not change significantly during further annealing. These are remarkably strong textures for a body-centered cubic metal compared e.g. to commonly reported high volume fractions of 30 % for Goss orientations in silicon steels. Reliable quantification of such strong texture components requires analysis of orientations individually.

1. Introduction

Tungsten is considered for plasma-facing components in future fusion reactors as armor material for the first wall and the divertor. Pure annealed tungsten has a high brittle-to-ductile transition temperature and behaves brittle at room temperature [1] restricting its use as plasma-facing material. On the contrary, plastically deformed tungsten behaves in general more ductile at ambient temperatures and large plastic deformation has been utilized for fabrication of ductile tungsten wires by wire drawing [2]. During operation as plasma-facing component at high temperatures, the deformation structure induced by plastic deformation becomes unstable. Restoration processes as recovery, recrystallization and grain growth will unavoidably alter the microstructure and impair the desired mechanical properties [3]. In particular, recrystallization will reinstate the intrinsic brittleness of tungsten. Several concepts have been pursued to increase the thermal stability of the microstructure or at least to control the microstructural changes by restricting the motion of grain boundaries. Such an impedance of grain boundary motion can be achieved by alloying tungsten with dispersed oxides or carbides pinning the grain boundaries (e.g. [4]). Alternatively, tungsten can be doped with small amounts of potassium; potassium is insoluble in tungsten and forms bubbles in the microstructure hindering grain boundary motion. Doping tungsten with potassium has been successfully applied in manufacturing of heavily drawn tungsten wires [2]. The same strategy has been adapted to commercially rolled tungsten plates. The texture evolution during annealing of cold-rolled tungsten sheets with a comparably high potassium content of 80 ppm is investigated in detail.
2. Materials and techniques

2.1. Material

Thin tungsten sheets are provided by PLANSEE SE. The material is doped with 80 ppm potassium – a higher content of potassium than commonly used in WVM (tungsten vacuum metallizing) material. The cold-rolled sheets with bright finish extend 180 x 30 x 0.1 mm$^3$ along transversal, rolling and normal direction (TD, RD, and ND), respectively. The sheets are cut by electro discharge machining into smaller pieces of 5.5 x 4.5 x 0.1 mm$^3$. For protection against oxidation and formation of volatile WO$_3$ during annealing, cut specimens are encapsulated individually in quartz glass ampoules. The ampoules are evacuated, flushed with Argon (with purity higher than 99.999%), evacuated again and sealed with a blowtorch. Specimens in their ampoules are put in a pre-heated ceramic tube furnace NaberTherm RHTC 80/230/15, removed after the desired annealing time and cooled to room temperature by air-cooling. Encapsulated specimens are annealed in an isochronal series for 2 h at different temperatures (800 °C, 1000 °C, 1100 °C and 1300 °C). An additional isothermal series is performed at 1300 °C for longer times (2 h, 24 h, 48 h, 96 h, 168 h, 336 h and 504 h).

2.2. Microstructural investigation

Individual specimens are embedded conductively sandwiched between two protective steel plates to avoid delamination. Cross sections containing transversal and normal direction are prepared for electron backscatter diffraction by mechanical grinding (subsequently on SiC-paper of grit sizes 500, 1000, 2000, and 4000) and mechanical polishing (with diamond suspension with grain size 3 µm). Due to delamination issues and severe edge effects caused by electro-polishing, chemo-mechanical polishing by an alkaline colloidal silica suspension (OP-S by Struers ApS) is applied as final step leading to suitable specimen surfaces. Orientation data are gathered by electron backscatter diffraction on freshly prepared cross sections using a Bruker NOVA NanoSEM 600 equipped with a Bruker e-Flash HD EBSD detector applying a voltage of 15 kV. Large, statistically representative maps through the entire thickness are recorded on the cross sections with a step size of 0.1 µm. Without any filtering or removal of non-indexed points, the gathered EBSD data are analyzed using the MTEX toolbox Version 5.5.0 (cf. [5]) and evaluated further by own purposely developed routines.

3. Results

3.1. Microstructure of the as-rolled condition

Figure 1 presents orientation maps obtained on the TD/ND section of specimens in the as-rolled condition. The maps are colored according to the crystallographic direction along the rolling direction (figure 1a) and the normal direction (figure 1b). The respective dominating green and red colors, reveal the preference of aligning <110> directions with RD ($\alpha$-fiber) and <100> directions with ND or, equivalently, {100} planes with the rolling plane (θ-fiber) – both typical fiber texture components after rolling of body-centered cubic (bcc) metals [6]. Only a small number of high angle boundaries are discerned in the orientation maps indicating that only minor orientation differences less than 15° exist between neighboring orientations.

To quantify the texture, the orientation distribution function (ODF) [7] is calculated from all individual orientations in the map using a de la Valle Poussin kernel [5] with a half width of 5°. Figure 2 shows the orientation density with respect to a random distribution of orientations in a section where the Euler angle $\phi_2$ is kept constant to be 45°. The ODF section reveals a strong preference of orientations with Euler angles ($\phi_1, \theta, \phi_2$) = (0°,0°,45°). This ND rotated cube orientation {100}<011> can be obtained by rotating an ideal cube orientation, (0°,0°,0°) or {100}<001>, around the normal direction by 45°. In figure 2a, one of the typical texture components after rolling of bcc metals [6], the $\gamma$-fiber with one of the {111} planes in the rolling plane, seems to be missing entirely. When choosing a different scaling for the orientation density as in figure 2b, the presence of $\gamma$-fiber components is confirmed, but only to a very small extend.
The 19th International Conference on Textures of Materials (ICOTOM19 2021)  
IOP Conf. Series: Materials Science and Engineering 1121 (2021) 012018  
doi:10.1088/1757-899X/1121/1/012018

Figure 1. Orientation maps of the TD/ND cross section of a thin cold-rolled potassium-doped tungsten sheet. The colors reflect the crystallographic directions along (a) the rolling direction and (b) the normal direction according to the inset. High angle boundaries with disorientation angles above 15° are marked in black, low angle boundaries with angles between 2° and 15° in white.

Figure 2. Orientation distribution function in the section $\phi_2 = 45°$ of a thin cold-rolled potassium-doped tungsten sheet with different scaling: (a) to the maximum orientation density of 114 and (b) to an orientation density of 3.5. All orientation densities are given in multiples of random distribution.

The texture strength can be quantified by the maximal orientation density, the texture index [7], or the texture entropy [8]. All three measures are listed in table 1. Additionally, the volume fractions for different fiber components typical for cold-rolled metals are determined. The values in table 1 and figure 3 depend on the half width of 5° used for calculating the ODF. Choosing a lower half width will result in even higher orientation density, texture index and fiber volume fractions for sharp textures.

Table 1. Quantitative texture analysis of thin cold-rolled potassium-doped tungsten sheets.

| Condition | Max. orient. density | Texture index (for resolution 1°) | Texture entropy | Volume fraction of fibers from ODF / % | Vol. fraction rotated cube / % from all orientations |
|-----------|----------------------|----------------------------------|-----------------|---------------------------------------|---------------------------------------------------|
| As-rolled | 114                  | 28.6                             | -2.28           | 82.4                                  | 9.6                                               |
| 1300 °C, 2 h | 201                  | 68.2                             | -3.53           | 95.6                                  | 0.3                                               |
| 1300 °C, 96 h | 194                | 65.4                             | -3.44           | 95.4                                  | 2.3                                               |
Further analysis of the texture is not based on the ODF to avoid artefacts of smoothing by the de la Valle Poussin kernel and its chosen half width. Instead all orientations in the map are tested individually. The deviation of an orientation from the ideal rotated cube orientation is evaluated by its disorientation angle. The volume fraction of the rotated cube component defined as the fraction of indexed points having disorientation angles not exceeding a chosen threshold value is illustrated in figure 4. Allowing larger deviations from the rotated cube orientation, a larger volume fraction is revealed. For threshold angles of 5°, 10° and 15°, volume fractions for the rotated cube of 19.2 %, 51.4 % and 67.0 % are obtained, respectively. This confirms that a strong rotated cube texture is present. For comparison, the corresponding numbers for the rotated cube orientation in a random distribution of orientations are 0.08 %, 0.67 % and 2.27 % [9].

3.2. Annealed conditions
Orientation maps after annealing for 2 h and 96 h are shown in figure 5 as an example. The dominance of the red color is even stronger than in the as-rolled condition indicating an even stronger preference for texture components of the θ-fiber. High angle boundaries are even less frequent than in the as-rolled condition. A preliminary texture analysis of the annealed conditions is based on texture index and the volume fraction of the characteristic fibers (as determined from the orientation distribution function). Table 1 reveals that the texture index (obtained with a resolution of 1°), which has a rather large value already in the as-rolled condition, becomes even higher during annealing and the texture sharpens. As seen from figure 3, the low volume fraction of γ-fiber components of about 10 % in the as-rolled condition is reduced by annealing to less than 1 %. This is confirmed by the observation of a single surviving band. The volume fraction of the θ-fiber texture component rises above 80 % at 1000 °C and even 90 % at 1300 °C (cf. figure 3); the strong α-fiber strengthens further. Due to the simultaneous strength of α- and θ-fiber, the most dominating orientation must be the rotated cube orientation {100}<011>.

The strength of the rotated cube component is further evaluated by an analysis of all individual orientations in the maps and their orientation difference from the ideal rotated cube orientation {100}<011>. Taking into account cubic crystalline symmetry, the disorientation angle between each orientation and the ideal rotated cube orientation is calculated. The accumulated probabilities (corresponding to volume fractions) for annealing at 1300 °C for 2 h and 96 h are illustrated in figure 4. For both annealed conditions much sharper distributions are obtained and large deviations from the rotated cube orientation are less likely than in the as-rolled condition. For instance, half of the orientations deviate less than 5.9° or 6.1° from the ideal rotated cube orientation after annealing at 1300 °C for 2 h or 96 h, respectively, compared to 9.7° in the as-rolled state.

The different volume fractions of the rotated cube component obtained on all annealed conditions are illustrated in figure 6 accepting a maximum disorientation angle of 15° from the {100}<011>

![Figure 3. Volume fraction of different fiber texture components in a thin cold-rolled potassium-doped tungsten sheet during isochronal annealing for 2 h at different temperatures.](image1)

![Figure 4. Volume fraction of the rotated cube texture component in dependence on the tolerated disorientation angle from the ideal {100}<011> orientation.](image2)
orientation. The rotated cube component, which already occupied a high volume fraction of 67.0% in the as-rolled state, remains almost unaltered upon annealing for 2 h at 800 °C. For annealing for 2 h at higher temperatures, the volume fraction increases with annealing temperature as seen from figure 6a. For the highest investigated temperature of 1300 °C, a volume fraction of 94.8% is reached after 2 h. During longer annealing at 1300 °C, the volume fraction does not increase further and remains almost constant (cf. figure 6b). Table 1 illustrates the effect of the chosen threshold angle for the allowed deviation from the ideal {100}<011> orientation. Clearly smaller volume fractions are determined, if only smaller disorientation angles are tolerated. Nevertheless quite significant volume fractions are resolved even for low angles as 5° and 10°. The apparent discrepancy of a larger volume fraction for 15° deviation from rotated cube than the θ-fiber volume fractions is explained by the difference in deriving volume fractions directly from individual orientations instead by means of the ODF – the latter leads to too low fractions for the rotated cube component allowing 15° deviation (cf. table 1).

![Figure 5](image1.png)

**Figure 5.** Orientation maps of TD/ND cross sections of thin cold-rolled potassium-doped tungsten sheets after annealing at 1300 °C for (a) 2 h and (b) 96 h. The colors reflect the crystallographic directions along the normal direction according to the inset. High angle boundaries with disorientation angles above 15° are marked in black, low angle boundaries with angles between 2° and 15° in white.

![Figure 6](image2.png)

**Figure 6.** Volume fraction of the rotated cube texture component (accepting a maximum disorientation angle of 15°) in thin cold-rolled potassium-doped tungsten sheets after annealing: (a) isochronally for 2 h at different temperatures and (b) isothermally for different times at 1300 °C.
4. Discussion
A rather strong rotated cube texture is present in cold-rolled potassium-doped tungsten sheets which increases even further during annealing. During the first 2 h of annealing at a temperature of 1300 °C, grains become slightly larger and less elongated (as seen comparing the boundaries in figures 1b and 5a), nevertheless the appearance of a deformed, yet coarsened microstructure is kept, ruling out primary recrystallization as restoral process. The observed coarsening is driven by elimination of high angle boundaries leaving a network of immobile low angle boundaries behind. The process must be addressed as extended recovery (in accordance with observations [10] that nucleation in bcc metals in the rotated cube component is indolent) and texture strengthening is achieved solely by minor local motion of high angle boundaries. No long-range motion of high angle boundaries is required; hence, the process cannot qualify as primary recrystallization [11].

The texture strength achieved by annealing is remarkable. Usually textures with much lower orientation densities and texture indices are considered strong (e.g. in rolled pure tungsten plates [12]). For face-centered cubic metal, a cube volume fraction of 96 % has been achieved for Ni-W substrates [13], whereas in the classical case for bcc materials, a volume fraction of Goss orientations \{110\}<001> of 30 % is considered high in hot-rolled silicon steels [14]. These volume fractions are obtained after optimizing the process for the desired texture. This is not attempted here yet and one may expect that even higher volume fractions of rotated cube orientation are achievable.

5. Conclusion
The texture of cold-rolled tungsten sheets with a high potassium content of 80 ppm and its evolution during annealing between 800 °C and 1300 °C is investigated. Electron backscatter diffraction reveals a strong rotated cube texture component (with volume fraction 67.0 %) in the as-rolled state, which strengthens during annealing for 2 h at 1000 °C and above. Annealing at 1300 °C causes a remarkably high volume fraction of 94.8 % not changing significantly with further annealing up to 504 h. All orientations must be considered individually to quantify such strong texture components.

Acknowledgments
This work has been carried out partially within the framework of the EUROfusion Consortium and has received funding from the Euratom research and training programme 2014-2018 and 2019-2020 under grant agreement No 633053. The views and opinions expressed herein do not necessarily reflect those of the European Commission.

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