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The crystallographic relationship of molybdenum textures after hot rolling and recrystallization

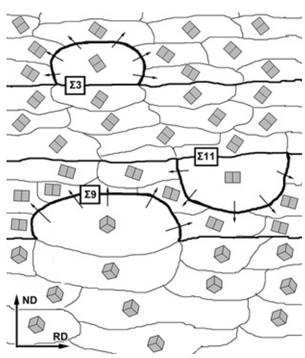
M.L. Lobanov [a,*], S.V. Danilov [a], V.I. Pastukhov [a,b,c], S.A. Averin [b,c], Y.Y. Khrunyk [a], A.A. Popov [a]

[a] Ural Federal University named after the first President of Russia B.N. Yeltsin, 19 Mira Street, Ekaterinburg 620002, Russia
[b] Institute of Nuclear Materials, Sverdlovsk Region, Zarechny 624250, Russia
[c] National Research Nuclear University «Moscow Engineering Physics Institute», Kashirskoe shosse, 31, Moscow 115409, Russia

HIGHLIGHTS

- The texture of molybdenum sheet has been shown to be characterized by a set of stable orientations across its whole width.
- During the recrystallization process (001)[110] weakens, while {112}⟨110⟩, {111}⟨112⟩ and {111}⟨110⟩ enhance.
- The orientations of recrystallization and deformation are connected by turns at specific angles around the ⟨110⟩-axes.
- The formation of recrystallization texture is due to the migration of Σ9 CSL and/or the formation of nuclei at the Σ3, Σ11.

GRAPHICAL ABSTRACT



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ABSTRACT

Using the method of orientation microscopy, based on electron backscatter diffraction (EBSD), the texture of molybdenum sheet, produced by hot rolling at 1100 °C with a total strain exceeding 90%, was characterized. Throughout its whole thickness, the molybdenum texture consisted of a set of stable orientations: (001)[110], {112}⟨110⟩, {111}⟨112⟩, {111}⟨110⟩. During recrystallization at 1200 °C {001}⟨110⟩ orientation became weaker, while the other orientations enhanced. The orientations of recrystallized grains were related with the orientations of deformed grains by rotations at defined angles around their axes ⟨110⟩. The formation of recrystallization texture can be explained by the migration of CSL boundaries of Σ9 type and/or the formation of recrystallization nuclei at the Σ3 and Σ11 CSL boundaries, which was preceded by the appearance of CSL misorientations between the components of deformed texture.

1. Introduction

Molybdenum's unique combination of properties (high melting point, high temperature strength, low vapor pressure and low coefficient of thermal expansion at elevated temperatures, high thermal and electrical conductivity) enables its application as a construction material for mechanisms and equipment, associated with the use at high temperatures in an oxygen-free atmosphere, in particular in the areas of nuclear power engineering and electrical industry. Besides, molybdenum is one of the candidate materials for high-temperature, gas-cooled space reactor pressure vessel [1].

* Corresponding author.
E-mail address: m.l.lobanov@urfu.ru (M.L. Lobanov).

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The crystallographic texture provides obtained products with improved physical-mechanical properties. The formation and development of texture occurs at the manufacturing stage and results from the deformation and heat treatment steps. The knowledge of texture evolution can be useful to optimize the processes of products manufacturing by the modification of deformation intervals along with temperature and time ranges. In products, made of alloys containing high-melting-point metals with body-centered cubic (bcc) lattice (such as α-Fe, β-Ti, Mo, W), the fracture of metals is associated with the local texture. In these metals the cracks propagate parallel to the crystallographic planes {100}. Furthermore, the development of cracks is largely dependent on the presence of extended areas having a specific texture (001)[110] throughout the whole length, which exceeds the critical size of the crack [2,3].

The structure and texture of molybdenum sheets, used as raw material to obtain different products, has been a subject for extensive research since many years [4–12]. It was shown that the rolling and primary recrystallization textures in molybdenum sheets were typical for free of impurities, bcc metals [4]. If the molybdenum sheet was rolled in one direction, its texture consisted of main orientations: (001)[110], {111}⟨112⟩ and {112}⟨110⟩, while for cross-rolled molybdenum sheets (001)[110] and {111}⟨112⟩ orientations were observed. When annealing was followed by recrystallization deformation texture was preserved [4]. Consistent results [4] were obtained by another research group [5], who explained the formation of deformation texture in terms of the relaxed constraints Taylor theory [5]. In two other studies [6,7], the cold rolling texture of a molybdenum sheet (ε ~ 60%) was simulated employing different plasticity models. The authors demonstrated the advantages of different models, which depend on the morphology of grain structures. Next, other researchers [8,9] revealed, that cross rolling affects the deformation texture of molybdenum sheets, transforming {112}⟨110⟩ orientations from α-fiber texture (axis ⟨110⟩ || RD (RD = Rolling Direction)) into {112}⟨111⟩ orientations, which proved to be unstable during further deformation. This led to some changes of the recrystallization texture and was in agreement with relaxed constraints Taylor theory. In the other study [10] Primig et al. analyzed the impact of heating rate (1–1000°/min) on the recovery processes and recrystallization behavior of hot rolled molybdenum. The researchers demonstrated that lower heating rates (1–100°/min) caused the development of recovery processes, which significantly slowed down recrystallization and resulted in increased volume fractions of recrystallized grains. Another group of researchers carried out an elegant study on the orientation dependence of the dislocation microstructure in molybdenum, deformed by the compression [11]. After the compression strain reached 40%, three types of microstructure of original grains were developed in an orientation-dependent manner: cells having dislocation boundaries and ⟨100⟩ axis parallel to the compression direction (type 2); cell blocks with extended planar boundaries parallel to a ⟨100⟩ crystallographic axis, deviated from the compression direction to an angle exceeding 30° (type 1); cell blocks with extended planar boundaries deviated from the compression direction to an angle exceeding 30° and having a ⟨110⟩ axis, approximately parallel to the compression direction. During further deformation, dislocation boundaries underwent more modifications of size and shape, which was explained by different slip systems within the grain orientations. Moreover, the previous studies [12] revealed that the texture of hot rolled molybdenum corresponded to the typical texture of bcc metals, which is represented by the complex of two components: a strong α-fiber and a weak γ-fiber (axis ⟨111⟩ || ND (ND = Normal Direction)). Recrystallization of γ-fiber components occurred faster. New grains were nucleated at the shear bands and the grains growth rate was quite slow. It was also shown [13–15], that the relationship between deformation and recrystallization orientations may be represented as a strictly crystallographic one and it can be determined by the formation of coincident site lattice (CSL) between the neighboring grains. Besides, it is necessary to note, that several studies pointed to the role of CSL boundaries in the formation of secondary recrystallization texture in bcc crystals (Fe – 3 wt.% Si alloy) [16–18].

Thus, according to almost all the existing models on the evolution of grain orientations during deformation including the Taylor theory, the formation of stable orientations is associated with high levels of strain [19]. Such stable orientations, having specific crystallographic indices, do not change their space orientation if further deformation occurs. The latter is due to the balanced slip systems having reverse directions. Under strain, following a specific deformation scheme, a set of discrete grain orientations exhibiting specific misorientations relative to each other is formed. Employing the crystallographic analysis of relative misorientations, within the deviation, defined by Brandon criterion, possible CSL misorientations can be revealed [15]. Presumably, the knowledge of the role of CSL boundaries in the formation of recrystallized grains orientations enables to predict the texture and the anisotropy of physical and mechanical properties of the product, which result from the deformation and heat treatment steps.

Our research aimed to analyze the texture state of molybdenum after hot rolling and annealing, study the crystallographic relationship between deformation and recrystallization orientations of grains and reveal a possible role of CSL boundaries in the process of structural transformation.

2. Materials and methods

The research was conducted using a 2 mm thick molybdenum sheet, produced by hot rolling at 1100 °C with a total strain exceeding 90%. Specimens were cut from molybdenum sheet and annealed in the vacuum furnace at 1200 °C for 45–300 min. Notably, 1200 °C corresponds to the temperature of the coolant adjacent to the reactor pressure vessel.

Surface morphology was analyzed by scanning electron microscope TESCAN Mira3 LMU at an accelerating voltage of 20 kV. To determine the orientation of grains and analyze the texture we employed EBSD HKL Inca attachment and the Oxford Instruments System software. The scanning step was 0.2 μm, the error in the determination of the crystal lattice orientation did not exceed ±1° (about ±0.6° on the average). The low-angle boundaries between local volumes were plotted on orientation maps at misorientation ranging from 2 to 15°. In figures, the thickness of the boundaries was equal to one pixel. At misorientation ≥15° high-angle boundaries were drawn with the boundaries thickness of three pixels (Figs. 1a; 2a; 3a). The images were analyzed at the cross sections. In central and sub-surface areas, sections with the size of 160×160 μm were investigated. The analysis of the texture of the sections with the size of 160×160 μm (Figs. 1b, c; 2d, c, d; 3b) was conducted employing the orientation distribution function (ODF) of the Oxford Instruments System software. The figures demonstrate ODF of sections with the most pronounced texture components. The Fig. 1e shows the locations of main texture components of the cold rolled bcc metals in transverse direction (TD). To analyze special boundaries between separate grains, orientation maps were constructed using the Oxford Instruments System software with the standard Brandon criterion. Each boundary has a specific ΔΘ value, denoting the deviation of experimentally measured misorientation from the exact coincident site lattice (CSL) misorientation, and which is derived from the equation $\Delta\Theta = 15°/(\Sigma n)^{1/2}$, where Σn denotes the number of coinciding sites in three-dimensional orientation mapping. For analyzed sections with the size of 160×160 μm, the frequencies of occurrence of CSL boundaries, characterizing CSL misorientations between the components of material texture, were also analyzed (Figs. 1d; 3c). In the orientation maps, which fixed the state of incomplete primary recrystallization (Fig. 2a), the separation of deformed and recrystallized fractions was conducted manually by operator. The deformed fractions differed from the recrystallized fractions by size, a more elongated shape in the rolling direction (RD), and a higher number of low-angle grain boundaries. Notably, in large recrystallized grains the value of local