Int. Journal of Refractory Metals and Hard Materials 60 (2016) 92–98

Contents lists available at ScienceDirect

Int. Journal of Refractory Metals and Hard Materials

journal homepage: www.elsevier.com/locate/IJRMHM

Cold rolled tungsten (W) plates and foils: Evolution of the microstructure

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ARTICLE INFO

Article history:
Received 29 March 2016
Received in revised form 17 June 2016
Accepted 29 June 2016
Available online 2 July 2016

Keywords:
Cold rolling
Severe plastic deformation (SPD)
Ultrafine-grained tungsten (UFG-W)
Rotated cube texture
High-angle grain boundaries (HAGB)
Microstructure

ABSTRACT

This paper is the first part of our series on ultrafine-grained (UFG) tungsten produced by cold rolling. The aim of our project is to investigate the correlation between microstructure, mechanical properties and deformation mechanisms in UFG tungsten, a material interesting for future high-temperature applications. For this purpose, a batch of tungsten sheets with different thicknesses has been produced by subsequent cold rolling out of a single sintered compact.

The aim of this paper is to characterise the microstructure of the as-received tungsten sheets. Quantitative grain size analysis by EBSD confirms a grain refinement by cold rolling well down into the UFG-regime, reaching 240 nm in the S-direction for the 100 μm foil. The grain refinement comes with an increase in the number of HAGBs, while LAGBs show no correlation with the degree of deformation introduced by cold rolling. All plates are distinctively textured, the main texture being the {001}⟨110⟩-orientation (rotated cube texture).

Vickers microhardness measurements confirm a steady increase in hardness with advanced rolling up to 687 kg/mm^2 for the 100 μm foil. This indicates that the one-dimensional grain refinement to UFG by cold rolling is sufficient to achieve properties comparable to other UFG tungsten specimens. Therefore, it can be concluded that the batch of samples introduced in this paper will be fit to investigate the evolution of mechanical properties and deformation mechanisms in correlation with the microstructure.

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1. Introduction

Progress in high-temperature applications demands new materials with enhanced thermomechanical and thermophysical properties, for future energy systems like fusion, or for optimising efficiency in existing applications by making higher temperature differences available. From a functional point of view, tungsten exhibits outstanding properties in oxygen-free environments, like the highest melting point of all metals, a high recrystallisation temperature, good thermal conductivity, as well as high temperature strength and creep resistance [1]. However its high brittle-to-ductile transition temperature (BDTT), resulting in brittle behaviour at low temperatures and a difficult manufacturing process, impedes its application as a structural material. Many efforts have been made to ductilise tungsten at lower temperatures [2,3], the three main approaches being alloying (e.g. with rhenium [4] or iridium [5]), forming tungsten composites (e.g. tungsten-fibre reinforced tungsten [6,7]) or tailoring the microstructure [8–10]. Influencing the microstructure down to smaller grain sizes offers the application of pure tungsten and promises the enhancement of the two main mechanical properties: strength and ductility. However, a purposeful application demands a deeper understanding of the deformation mechanisms in tungsten and their dependence on the microstructure.

Several studies on single crystal tungsten have helped to develop a deeper understanding of the brittle behaviour, the low BDTT and the high strain rate sensitivity (SRS) by identifying the bcc-typical screw dislocation with a three-fold symmetric dislocation core and its movement by kink-pair formation as the controlling mechanism of plastic deformation [11,12]. Less is known of polycrystalline tungsten, where the microstructure with its texture and deformed grains adds more complexity [13]. With the rise of nanotechnology, it became possible to create a microstructure with grains smaller 1 μm, thus adding intrinsic size effects to the deformation mechanisms [14].

Different approaches to produce ultrafine-grained (UFG) tungsten are known: Mathaudhu [15] achieved a UFG grain structure by equal channel angular pressing (ECAP), while Wei [14] showed the suitability of ECAP followed by subsequently rolling at decreasing temperatures. Furthermore, Wei introduced the concept of low-temperature rolling a sintered compact of commercial tungsten to produce UFG tungsten [16]. “Low-temperature rolling” or also “cold rolling” is used for rolling at temperatures under 1000 °C (1273 K), therefore well below the recrystallisation temperature of tungsten. The cold rolling of tungsten

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http://dx.doi.org/10.1016/j.ijrmhm.2016.06.020
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results in a pancake-shaped, layered laminar structure [17,18]. Wei [16,19] and Kecskes [9] were able to show that, compared to coarse grained tungsten, tungsten with a UFG microstructure exhibits higher strength and ductility, a reduction in strain rate sensitivity (SRS), a transition in deformation mode to localised shearing and an elastic nearly-perfectly plastic stress-strain-behaviour (i.e. no strain hardening). Furthermore, a significant toughness and a decrease of the BDTT were demonstrated by Faleschini and Pippan [8], Németh et al. [20] and Reiser et al. [21] for thin cold rolled foils, validating the potential of tungsten for use in structural applications. However, "despite a few interesting findings, a systematic study of the various aspects of plastic deformation of severe plastic deformation (SPD) processed ultrafine-grained tungsten is still lacking" [16]. Within this paper we present a systematic study to elucidate the issues mentioned above.

A batch of tungsten sheets with different thicknesses has been produced by subsequent cold rolling to shed light on the deformation mechanisms in UFG tungsten and to access the question of whether the good properties in tungsten foils result from the ultrafine grain size or are already found in cold rolled plates with coarser grains due to cold rolled-introduced effects. All plates are rolled out of the same sintered compact of technical pure tungsten (99.97 wt% W). This unique batch of samples allows us to investigate the mechanical properties without the influence of chemical composition or fabrication differences in the production of the sintered compact. The mechanical properties can therefore be correlated directly to the grain size and cold work induced lattice defects.

For this purpose, three main aims are projected: (1) characterisation of the microstructure and defect structure of the as-received plates, (2) determination of the mechanical properties and deformation mechanisms indirectly by mechanical testing as tensile- and strain-rate-jump-tests, (3) identification of deformation mechanisms directly by high-resolution electron microscopy. In this paper, point (1) should be treated by a basis analysis. The following sections will address the questions:

(1) How is the evolution of the grain size related to increasing deformation (thinner plates), and do severe cold rolled tungsten foils have an ultrafine-grained microstructure?
(2) How do high-angle grain boundaries (HAGBs), low-angle grain boundaries (LAGBs) and texture evolve?
(3) Do the different plates show a change in hardness, hinting at a change in mechanical properties due to the microstructure?
(4) Does the chemical composition change through cold rolling and can further investigation of mechanical properties be attributed solely to the evolution of the microstructure?

To address these questions, the paper is organised as follows: The as-received material and its fabrication as well as the characterisation methods are briefly described, followed by the display and discussion of the results of the basis analysis. Finally, the conclusions of the results will be summarised with respect to further testing of the material, the aim being to access the deformation mechanisms.

2. Materials and methods

2.1. Cold rolling

The material analysed here is a series of tungsten sheets with different thicknesses produced by PLANSEE SE in Reutte, Austria. The sheets are rolled out of a single sintered compact of commercially pure tungsten, hence promising the same chemical composition of all sheets and leaving the microstructure as the only factor influencing the mechanical properties. The sintered compact has a density of approximately 94% and consists of equiaxed grains with an average grain size of 18 μm.

Due to the poor workability of tungsten, the rolling has to be performed at elevated temperatures. However, the high melting point of tungsten and the consequential high recrystallisation temperature allows for rolling up to 800–1000 °C (1073–1273 K) without a recrystallisation process counteracting the desired grain refinement [15,17]. The tungsten sintered compact is heated in a first step to a temperature of above 1250 °C (1523 K) ("hot-rolling") and is subsequently rolled to a thickness of 5.5 mm, annealing it between the rolling steps. After heating this plate to a temperature of below 1000 °C (1273 K) ("low-temperature-rolling"), it was then brought down by progressive rolling to thicknesses of 1 mm (true strain $\varphi = 1.7$), 0.5 mm ($\varphi = 2.4$), 0.3 mm ($\varphi = 2.91$) and 0.2 mm ($\varphi = 3.31$). At each of the mentioned steps, one piece was kept for further testing. In a final step, a 100 μm thin foil ($\varphi = 4$) is produced after pre-heating to a temperature below 300 °C (573 K) ("cold rolling"). The rolling parameters are shown in Table 1. Furthermore, all received sheets experienced a stress-relieved annealing. From these plates, samples were prepared using wire-cut electrical discharge machining (EDM).

2.2. Chemical analysis

A chemical analysis was performed to check the purity grade of 99.97 wt% W and the maximum concentration of chosen elements. These elements comprise P, S, C, O, N as typical segregation elements at grain boundaries, as well as Fe, Cr, Mo, Si, Mn and V, which are possible impurities regarding the raw material and fabrication process. As a benchmark for all five plates, the 1 mm plate and the 100 μm plate were analysed to investigate the influence of the rolling procedure. For a quantitative analysis, a CS-analyser was used for C and S, carrier gas hot extraction (CGHE) for O and N and inductively coupled plasma optical emission spectroscopy (ICP-OES) for the remaining elements. Furthermore, a complementary Auger Electron Spectroscopy (AES) allows a surface-sensitive analysis to evaluate whether changes in chemical composition are due to surface contamination.

2.3. EBSD

The investigation of the microstructure is performed using a Zeiss Merlin field-emission-gun scanning electron microscope (SEM) equipped with an EDAX Hikari high-speed electron backscatter diffraction (EBSD) camera. Imaging the edges of the plates allows us to investigate the microstructure in the S-direction, which is the direction perpendicular to the rolling direction and therefore the most strongly altered by rolling. Analysis of EBSD datasets allows the identification of the grain size distribution, misorientation angles and texture. The analysis was performed using an acceleration voltage of 20 kV and approximately 10 nA probe current. An area of 30 × 24 μm with a step size of 20 nm was chosen. Points with a confidence index (CI) lower than 0.1 were removed during post-processing. Apart from a Grain CI Standardisation, no clean-up of the datasets was performed. The orientations of the data points remained unchanged. Misorientations between 5°–15° were considered low-angle boundaries; misorientations exceeding 15° were considered high-angle boundaries. Inverse-Pole-Figure-Maps are displayed with respect to the orthogonal sample directions.

Table 1
Rolling parameters and deformation degree (true strain).

Thickness [mm]	Sintered compact	5.5	1	0.5	0.3	0.2	0.1
φ_{TOTAL}	/	/	1.7	2.4	2.91	3.31	4
$T_{ROLLING}$	/	Hot rolling	Low-temperature-rolling				Cold rolling

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