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Microstructure and helium irradiation performance of high purity tungsten processed by cold rolling

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ABSTRACT

This work aims to investigate the effects of confined cold rolling on the evolution of microstructure, hardness, and helium irradiation performance of high purity tungsten (W). Using a final rolling temperature of 450 °C, W samples were severely deformed by confined cold rolling up to equivalent strains (ε_{eq}) of 1.6 and 3.3. Experimental results indicate that the average grain size of W specimens processed by confined cold rolling has been greatly reduced, and the rolled W samples with ε_{eq} ~3.3 do not show an "ideal texture" of (001)[110] which is the expected texture of bcc metals processed by conventional cold rolling. The irradiation resistance against 60 keV He^+ ions with up to a dose of 1.5×10^{22} ions·m^{-2} of the rolled W is compared to that of the as-received W. Results show that, due to an improvement of the metal's ductility, blister bursting with a partially opened lid forms on the surface of the rolled W, whereas blister bursting with a fully opened lid forms on the surface of the as-received W.

1. Introduction

Tungsten (W) has been considered as one of the most promising candidates for plasma facing materials (PFMs) for the first wall and divertor in the ITER (International Thermonuclear Experimental Reactor), EAST (Experimental Advanced Superconducting Tokamak), and other fusion reactors [1]. Fundamental research about the preparation and characterization of W-based PFMs with excellent mechanical and irradiation resistance properties has long been a subject of extensive efforts in the past several decades.

High performance PFMs are critical for the future success of the proposed fusion reactors, which will subject the PFMs to unprecedented flux levels of high energy neutrons along with an intense flux of hydrogen isotopes and helium ions. Advanced materials can enable improved reactor performance; excellent mechanical properties, with plasma radiation damage resistance, in particular, are, therefore, under intense investigation worldwide. In general, previously used key strategies for designing W-based PFMs with excellent mechanical performance and radiation resistance are mainly based on three methods: (1) alloying of W with Ti, V, Ta, or Re; (2) alloying of W with ultrafine grained and nanocrystalline refractory materials such as oxide dispersions and TiC; (3) carefully controlled and introduced alloying materials over a high density of interfaces or grain boundaries (GBs). These approaches have been summarized in Refs. [2–4].

Alternate to these techniques, a large volume of research has demonstrated the beneficial aspects of reducing the grain size to the ultrafine and nanoscale on material properties. Specifically, in recent years, nanocrystalline W (NC, grain size d < 100 nm) [5–7], ultrafine grained W (UFG, 100 nm < d < 1000 nm) [7,8] and multilayered materials [9–13] have been fabricated by severe plastic deformation methods including high pressure torsion (HPT) [5], equal-channel angular pressing (ECAP) [14], and accumulative roll-bonding (ARB) [15]. Obviously, the versatility and practical application of these methods open alternative and innovative avenues to tailoring the material performance; as such, they are appropriate to study the underlying grain size dependent mechanisms during plastic deformation. Recent results have shown that both mechanical and electronic properties (i.e., high strength and high conductivity) and improved radiation resistance of such materials are achieved because of the introduction of a high density of

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GBs and interfaces, which are sinks for irradiation-induced defects. Defect sinks also facilitate the recombination of the implanted ions and irradiation induced defects [16]. Although these fabrication routes are a means to produce such type of materials, they still have a fundamental challenge for industrial applications: only small samples can be prepared.

The mechanical property and irradiation resistance of coarse-grained W with average grain size of several tens of μm have been systematically investigated. More specific to our interests, recent research efforts focusing on finer W, in particular, UFG W with average grain size smaller than 500 nm, but greater than 100 nm, and NC W, with average grain size smaller than 100 nm, have been reported [6]. The use of UFG and NC W with high angle GBs have also been reported as one of the proposed solutions to mitigate helium induced radiation damage [7]. Notwithstanding the wealth of information described in these references, the mechanical and irradiation resistance of W with grain size from 500 nm to several μm is still lacking.

Fine-grained, oxide dispersion strengthened (ODS) W-based materials with a grain size from a sub-micrometer range to several micrometers have been fabricated by mechanical alloying and spark plasma sintering [17]. However, unfortunately, such methods suffer from multiple impurity contamination issues. The simplest way to prepare such fine-grained W without changing its chemical composition could be cold rolling (below the nominal recrystallization temperature). Moreover, it has been shown that the mechanical properties of W can be greatly improved by cold rolling. For example, the ductility and strength of commercial purity W have been greatly enhanced by cold rolling according to Wei and coworkers [18]. Furthermore, Reiser and coworkers [19,20] showed that a W foil with thickness of 0.1 mm is ductile under tension at room temperature. Recently, Zhang and coworkers [21] have investigated the basic thermo-mechanical properties and texture evolution of pure W with various hot rolling reductions. They concluded that the rolled W with 80% thickness reduction exhibited the best irradiation resistance. Nevertheless, a systematic study of the effects of confined cold rolling on the irradiation resistance of pure W is still lacking.

In this effort, the evolution of microstructure and microhardness of high purity W as a function of rolling thickness reduction was investigated. The microstructural evolution analyses were carried out via X-ray diffraction (XRD) analysis, electron backscattered diffraction (EBSD), scanning electron microscopy (SEM), and transmission electron microscopy (TEM). The mechanical properties were investigated by micro-indentation and nano-indentation measurements. Irradiation resistance of the rolled W was also evaluated by 60 keV He^+ ions with a fluence of 1.5×10^{22} ions·m^{-2}.

2. Experimental details

2.1. Material and sample preparation

The commercial W with purity of 99.99% was purchased from Advanced Technology & Materials Co., Ltd (AT&M; Beijing, China) and was used as the starting material. W pieces with dimensions of 8 mm × 8 mm × 15 mm were cut from the as-received plate using wire electrical discharge machining (EDM). The sharp edges of the W pieces were removed to avoid cracking during subsequent rolling. The work piece was sealed in a stainless steel canister of thickness of 0.3 mm to minimize oxidation and to prevent cracking. The as-received coarse-grained W specimen was designated as Sample-A for comparison. Confined cold rolling was performed on a laboratory rolling mill by progressively decreasing the separation between the two drums with a decrement of 0.01 inch (0.254 mm) per pass until the desired final thickness reduction was reached. In brief, the work piece was rolled from 8 mm to 6 mm at 850 °C, and then to 1.5 mm with progressively lowering the temperature to the final temperature of 450 °C. This sample was designated as Sample-B. The rolled sample was then retrieved from the stainless steel canister, and cut into small pieces for subsequent examination. The equivalent strain (ε_{eq}) corresponding to the total thickness reduction of Sample-B is ~1.6. The ε_{eq} was calculated by the following equation,

$$\varepsilon_{eq} = |ln(h/h_0)|, \quad (1)$$

where h is the thickness at a given deformation and h_0 is the initial thickness of the work plate.

One work piece that was rolled to 1.5 mm was sealed into a new 1-mm thick stainless steel canister and then further rolled to the final thickness of 0.3 mm at 450 °C. The wall of the stainless steel canister was thick enough to limit the deformation along the transverse direction (TD). This sample was designated as Sample-C. Finally, the rolled sample was retrieved from the stainless steel canister, and cut into small specimens for subsequent examination. The total thickness reduction of Sample-C was 96.25%, corresponding to a total equivalent strain of ~3.3. Since the melting point of W is 3687 K, its nominal recrystallization temperature is usually taken to be ~1200 °C. Therefore, the rolling in the present work may be considered as cold rolling, since the rolling temperatures were much lower than the recrystallization temperature of W.

2.2. Microstructural examination

The microstructure of the starting W sample was examined by optical microscopy along the TD after mechanical and electrochemical polishing. Electrochemical polishing was performed at room temperature using a NaOH solution (2 wt%) at 10 V DC. The cross sectional microstructures of the W samples rolled to ε_{eq} ~1.6 and ~3.3 were examined using SEM (JEOL-6480; JEOL [Beijing] Co., Ltd., Beijing, China). EBSD (MERLIN Compact; Carl Zeiss Shanghai Ltd., Beijing, China) was performed on the RD × TD plane of the rolled W sample with ε_{eq} ~3.3, where RD is the rolling direction. The sample for EBSD analysis was first polished up to 4000 grit emery paper, and then fine polished to a mirror finish using finer diamond paste. Finally, electrochemical polishing using the above mentioned electrolyte was performed on this rolled sample at room temperature. The CHANNEL 5 software developed by HKL Technology (Hobro, Denmark) was used to analyze the EBSD results. Misorientation angles less than 2° were excluded from the data during the calculation of the average grain size.

XRD was carried out on a Rigaku (Beijing, China) D/MAX-TTR III (CBO) diffractometer using Cu Kα radiation operated at 40 kV and 200 mA. The intensity from Cu Kα2 was stripped off using the Lorenz function. A theta-2theta configuration from 30° to 90° with a step size of 0.02° was used.

XRD Bragg profile analysis was used to analyze the dislocation density and dislocation character, based on the classical Williamson-Hall model [22] and the modified Williamson-Hall plot procedure [23]. However, within this 2theta range, there are only two obvious diffraction peaks for the rolled sample with ε_{eq} ~3.3, as will be shown in a subsequent section. As such, it is not accurate enough to determine the dislocation density by the above mentioned model. Instead, a single peak analysis using a simplified breadth method was used to determine the micro-strains of those samples. In the simplified breadth method, the XRD line profile can be approximated by a convolution of a Cauchy and a Gaussian function [24], where the Cauchy component is due to the small grain size effect and the Gaussian component is due to the micro-strain. In the present study, the effect of the small grain size on