Journal of Nuclear Materials 480 (2016) 281–288

Contents lists available at ScienceDirect

Journal of Nuclear Materials

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

Nanocrystalline-grained tungsten prepared by surface mechanical attrition treatment: Microstructure and mechanical properties

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ARTICLE INFO

Article history:
Received 5 May 2016
Received in revised form
24 July 2016
Accepted 26 August 2016
Available online 30 August 2016

Keywords:
SMAT
Tungsten
Microstructure
Nanograins
Dislocations
Mechanical properties

ABSTRACT

A nanostructured surface layer was fabricated on commercial pure tungsten using the method of surface mechanical attrition treatment (SMAT). The microstructure evolution of the surface layer was characterized by using scanning electron microscopy (SEM) and transmission electron microscopy (TEM) and its formation mechanism was discussed as well. Both refinement and elongation of the brittle W grains were confirmed. The elongated SMATed W was heavily strained, the maximum value of the strain at the grain boundaries reaches as high as 3–5%. Dislocation density in the SMATed W nanograins was found to be 5×10^{12} cm^{-2}. The formation of the nanograins in the top surface layer of the W was ascribed to the extremely high strain and strain rate, as well as the multidirectional repetitive loading. Bending strength of commercial W could be improved from 825 MPa to 1850 MPa by SMAT process. Microhardness results indicated the strain range in SMATed W can reach up to 220 μm beneath the top surface. The notched Charpy testing results demonstrated that SMATed W possess higher ductility than that of commercial W. The top surface of the W plates with and without SMATe processing possesses residual compressive stress of about −881 MPa and −234 MPa in y direction, and −872 MPa and −879 MPa in x direction respectively. The improvement of toughness (DBTT shift) of SMATed W may be the synergistic effect of residual compressive stress, dislocation density improvement and microstructure refinement induced by SMAT processing. SMAT processing could be a complementary method to further decrease the DBTT value of tungsten based materials.

1. Introduction

Nanostructured (NS) materials have been widely studied over the past couple of decades, since the decreasing grain size often suffer from undesirably high strength, as predicted by the well-know Hall-Petch relationship. However, NS materials generally possess poor ductility in spite of the ultrahigh strength. Processing artifacts (e.g. porosity) and lacking of strain hardening were considered to be main reasons for the low ductility of NS materials [1]. In the last few years significant advances were achieved in improving their ductility without losing the high strength in many materials by dislocation-mediated deformation [2]. However, in the case of intrinsic brittleness materials, it is difficult to store more dislocations in grains and continue the grain refinement.

As an important intrinsic brittleness material, tungsten (W) is currently attracting the greatest attention among materials for using as plasma facing materials in future fusion power reactors, because of its high thermal conductivity, high strength at elevated temperatures, low sputtering yield and low tritium inventory [3–5]. However, W based bulk (thick) materials exhibit serious embrittlement in several regimes, such as low-temperature

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http://dx.doi.org/10.1016/j.jnucmat.2016.08.032
0022-3115/

brittleness, high-temperature or recrystallization brittleness and radiation reduced brittleness and hardness [4]. Many methods have been employed to refine the grains to ultrafine-grained (>100 nm) or NS regimes (<100 nm) to improve the mechanical properties or irradiation resistance performance of the W [6], such as powder metallurgy (PM), severe plastic deformation (SPD) (including equal-channel angular extrusion (ECAE) [7,8], high pressure torsion (HPT) [9]) and surface machining [10].

In this study, we first use surface mechanical attrition treatment (SMAT) technique [1,11] to refine the surface grains into NS regimes in the bulk W at room temperature. The extremely large strain and strain rate induced by the SMAT process are expected to store more dislocations in the intrinsic grains at room temperature and refine the W grains into nanograin regime, thus improving the ductility without losing the strength. Meanwhile, the nanostructure (grain boundary) and dislocations could be an effective way to enhance radiation tolerance [3,12]. Besides, the gradient structure (from nano grains to submicro and micro grains) along the depth from top surface to strain-free matrix induced by SMAT process provides a unique opportunity to investigate the grain refinement mechanism by examining the microstructure features at different depths of the deformed surface layer [2].

2. Experimental procedure

The material used in this investigation was a rolled W plate (120 mm × 100 mm, and 4 mm in thickness), commercial purity. The typical microstructure of the commercial rolled W plate was revealed by optical micrograph (OM), as shown in Fig. 1(a),(b), the grain size is in a range of 25–60 μm. The set-up and procedures of the SMAT were described in elsewhere [1]. In this study, the SMAT to the W was performed under vacuum at room temperature for 15 min with a vibrating frequency of 20 KHz, using φ1.5 mm ZrO_2 ball. Surface microstructures at various depths were characterized by cross-section imaging, utilizing a Zeiss Ultra 55 dual-beam focused ion beam (FIB) scanning electron microscope. Cross-sectional TEM lamella was prepared using FIB. The plane-view TEM lamella of the layers from different depths were obtained first by polishing the corresponding surface layer and then and finally thinning by ion milling. All TEM samples were taken from the same SMATed sample. Transmission electron microscopy (TEM) observations were carried out on JEOL 2010 in PSI institute, Switzerland (Fig. 3, FIB prepared), and FEI Tecnai F20 microscopes (other TEM figures, ion milled), all operated at 200 kV.

Three point bending (3 PB) specimens of 34 mm × 4mm × 3 mm (length × width × height) were produced to measure the bending strength. Vickers microhardness tests were performed on the polished surface under a load of 200 g for 15 s. For Charpy impact tests, specimens were prepared based on the EU standards DIN EN ISO 148-1 and 14556:2006–10 with dimensions of 27 mm × 4 mm × 3 mm (length × width × height), a notch depth of 1 mm and a notch root radius of 0.1 mm. All Charpy impact tests sample were notched, and prepared in the L-S direction [23] ("L" stands for longitudinal, which means in the rolling direction, "S" stands for short transverse, which is the direction of the thickness of the plate). All samples were cut by electrical discharge machining. The specimens were heated to 600 °C, 700 °C, 750 °C, 850 °C and 900 °C in argon atmosphere, then pushed to the support outside the furnace and hit by a striker (25 J) immediately. For each test temperature three samples was tested. In fact, the heated specimens were exposed to the air in a very short time period. But the contact time is so short that the effect of air can be ignored. Residual stresses were measured in the directions perpendicular to the SMATed surface of W using a μ-X360n portable X-ray residual stress analyzer (Pulstec Industrial Co., Ltd.)

3. Results and discussions

3.1. Microstructure analysis

The cross-sectional microstructures of the SMATed W sample are shown in Fig. 2. It can be seen that the gradient structure resulting from a gradual decrease in the applied strain with increasing depth of the deformed layer, from very high (top surface) to zero (substrate), represents the entire range of the structure changes during the treatment [13]. The grain size of the treated W is lowest on the surface and increases towards the inner parts of the W. Elongated W grains can be observed closest to the surface, as shown in Fig. 1.

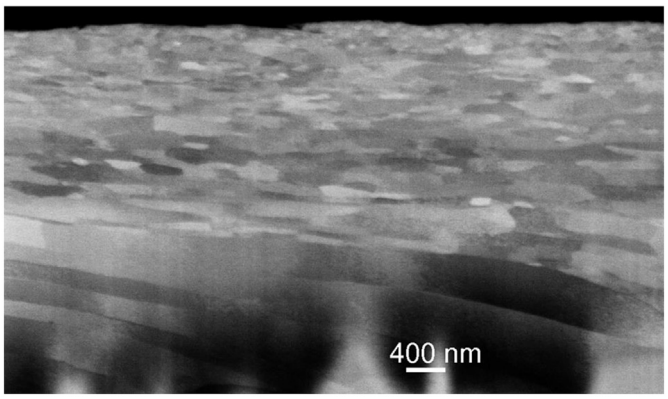

Fig. 2. Cross-sectional microstructures of the SMATed W sample with different magnification.

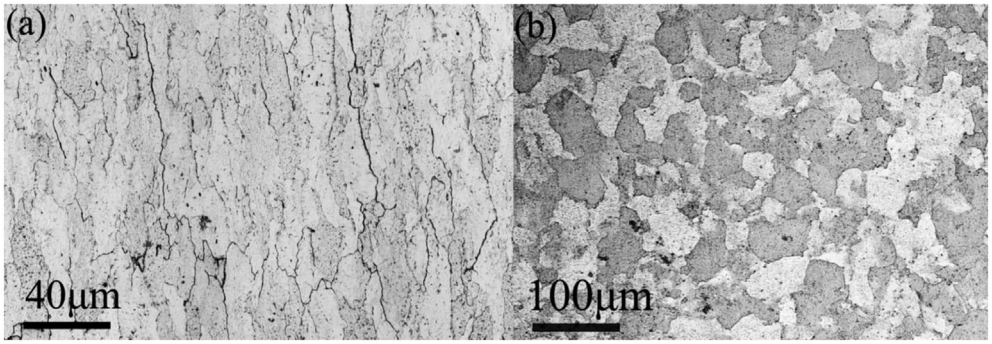

Fig. 1. Optical micrograph of commercial rolled W plate, (a) cross-sectional image, (b) top surface view. The grain was elongated in a range of 25–60 μm.