Int. Journal of Refractory Metals and Hard Materials 50 (2015) 184–190

Contents lists available at ScienceDirect

Int. Journal of Refractory Metals and Hard Materials

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

Temperature and deformation effect on the low and high angle grain boundary structure of a double forged pure tungsten

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ARTICLE INFO

Article history:
Received 6 November 2014
Received in revised form 12 January 2015
Accepted 20 January 2015
Available online 21 January 2015

Keywords:
Nuclear fusion
Tungsten
EBSD
Subgrain

ABSTRACT

In order to improve the performance of tungsten, a basic understanding of the microstructure–property relationships is essential. In the present study, a newly developed double forged pure tungsten grade from Plansee SE was investigated. By analysing the mechanical properties and microstructures in well-defined directions in the double forged tungsten, their relationships could be successfully correlated. A large amount of sub-grains with a typical size below 5 μm were observed in the as-received double-forged tungsten. After thermally treating the double forged tungsten up to 2000 °C, microstructural recovery was observed with the onset of recrystallization. Meanwhile, the sub-grain misorientation angle increased accompanied by sub-grain growth. The deformation temperature and the strain rate considerably influenced the final microstructure. The higher the temperature, the lower the amount of sub-grain boundaries due to sub-grain coarsening and the clearer the grain boundaries. The higher the deformation strain rate during tensile testing, the higher the grain orientation spread and the larger the sub-grain misorientation, but the smaller the grain size due to a lower extent of crystallization. This matched well with the mechanical testing data.

1. Introduction

Tungsten is being considered as the plasma facing material of the next step fusion devices such as the divertor for the international thermonuclear experimental reactor (ITER) [1]. The main advantages of W are its high melting point, high thermal conductivity, low thermal expansion, high resistance against sputtering and erosion and low tritium retention [2]. Different W grades produced by powder metallurgy, casting, plasma spraying, and chemical vapour deposition are industrially available [3]. Since these materials have different thermo-mechanical properties, their performance at the expected heat flux conditions could be significantly different. For fusion application, the most essential properties are thermal conductivity, ductility, structural stability at elevated temperature, and stability of properties under neutron irradiation and activation [4].

The main drawback of tungsten is its brittleness. Different methods have been tried to improve the toughness of tungsten [5–8], which also requires a balance of strength and ductility. For instance, dopants such as potassium are used to increase the recrystallization temperature of the tungsten [8]. Improvement of the fabrication technique is an alternative way. In the present study, the tungsten was double forged. The operational window of the ITER divertor [9] is limited by the rather high DBTT (ductile to brittle transition temperature) and low recrystallization temperature of tungsten.

To explain the high temperature tensile behaviour of testing bars machined out of the double forged material in different directions, as reported elsewhere [10,11], a deeper understanding of the inhomogeneity of the material at micro-meter level was essential. All material properties and microstructural evolution processes depend on the nature of the grain boundaries, such as their energy, mobility, and chemistry [12]. Therefore, the grain size and orientation as well as the sub-grain size and misorientation of the double forged tungsten are studied by electron backscatter diffraction (EBSD) in the present paper. Both the effect of temperature and structural changes caused by deformation were investigated by tensile testing of specimens at various temperatures in the 300–2000 °C range and subsequent analysis.

2. Material and testing procedure

The double forged pure tungsten (purity of 99.7%) was supplied by Plansee SE, Austria. The main impurities, as reported by the manufacturer, of this material are listed in Table 1. The production route for this material, consists of five steps; (1) cold isostatic pressing (CIP) of the tungsten powder, (2) hydrogen sintering at 2000–2500 °C, (3) hot forging of the sintered cylinder in the radial direction into a rod of 80 mm diameter which is subsequently cut to a length of ~140 mm, (4) hot forging along the cylinder axis into a flat disc shaped geometry (∅ =

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http://dx.doi.org/10.1016/j.ijrmhm.2015.01.008
0263-4368/

Table 1
Impurity content of the double forged pure tungsten.

Impurities	Ag	Ba	Co	Fe	Mn	Ni	Ti	Mo	Co	O	Si	Al	Ca	Cr	K
[μg/g]	10	5	10	30	5	5	5	100	30	20	20	15	5	20	10
Impurities	Na	Pb	Zn	H	Pb	As	Cd	Cu	Mg	Nb	Ta	Zr	N	S	
[μg/g]	10	5	5	5	20	5	5	10	5	10	20	5	5	5	

140 mm, height = 45 mm); and (5) removal of residual stresses by a thermal treatment at 1000 °C. Fig. 1a schematically presents the last three steps of hot forging and thermal treatment. Fig. 1b shows a picture of the as-received double forged tungsten disc.

The aim of this 5-step approach was to use a densification process yielding an isotropic material to be suitable as reference material on which all relevant thermo-physical and thermo-mechanical properties could be determined. However, in contrast to the assumptions, the material is only fully densified at the edge and there is a gradual increase in porosity up to 2% towards the centre [13]. Moreover, the double-forged material showed anisotropy in thermal shock behaviour as well as mechanical properties. The elongation at 500 °C in the transverse direction for example was higher than in the longitudinal direction [11].

Tensile testing was performed in a high temperature vacuum furnace (INSTRON) on dog-bone shaped samples (see Fig. 1c) with an overall length of 26 mm, a gauge length of 15 mm and an effective cross-section of 3×3 mm^2 machined out of the as-received cylinder in the longitudinal (= axial L) and transverse (= radial T) directions. The tests were conducted up to 2000 °C at loading rates of 0.2 mm/min and 42 mm/min [8]. Specimens for the tensile tests discussed in this paper were taken from the edge of the block in order to avoid porosity in the samples. As shown in Fig. 1c, the specimens for EBSD observation were taken either from a location near the fracture surface in order to study the combined temperature and deformation effect, or taken far away from the fracture surface to study the temperature effect. Additionally, pristine samples from the edge of the original block were used as reference.

All sample surfaces were prepared by grinding and followed by polishing (abrasive: diamond particles of 1 μm). In order to remove polishing deformation from the surface, electrolytic polishing was performed on a Struers Electropol polishing unit using a freshly prepared 4 wt.% NaOH solution in water. A polishing voltage of 15 V DC was applied for less than 2 min. The tungsten sample was used as anode and graphite as cathode using deionised water for cooling.

Electron back scatter diffraction (EBSD) or orientation imaging microscopy (OIM) measurements were performed on a scanning electron microscope (SEM model FEI-XL-30) with an EBSD detector. The patterns were recorded by a high-speed digital camera under a tilting angle of the sample stage of 60°. The acceleration voltage was 20 kV, spot size of 6 μm and scanning step size of 0.5–1 μm. Magnification for all the scans was usually maintained at 120× so that more than 500 grains were covered. The raw data obtained in these scans were processed with TSL OIM 5.31 pattern analysis software to obtain various information such as, grain size & distribution, grain orientation spread (GOS), micro-texture and grain boundary character distribution (GBCD).

3. Results and discussions

3.1. Sub-grain formation during double forging

The EBSD images of the as-received tungsten material taken in the longitudinal (L) and transverse (T) directions of the big block are presented in Fig. 2. In these inverse pole figures (IPF) or crystal orientation maps, the three main crystallographic orientations of the body-centred

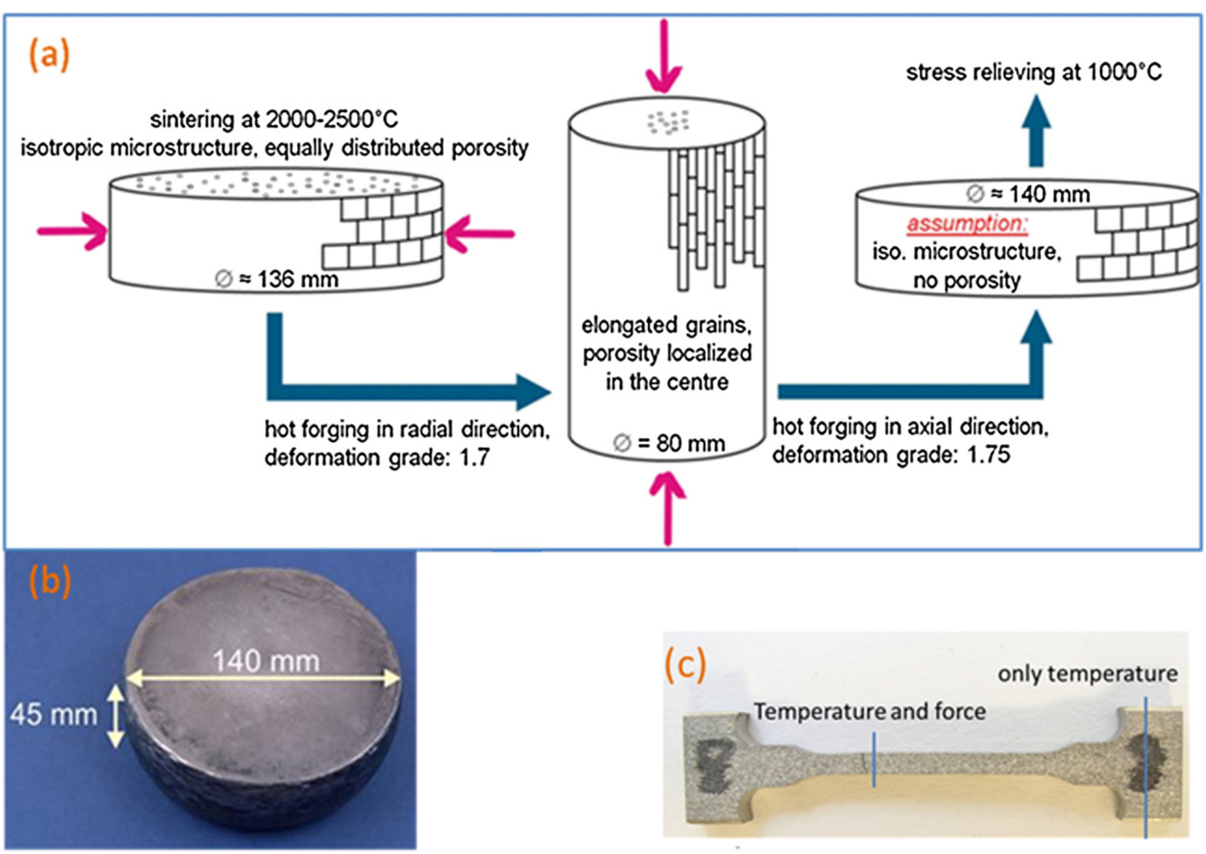

Fig. 1. (a) Schematic description of the material production process by sintering and double forging. (b) As-received tungsten block. (c) Tensile tested specimen with indication of the locations that were investigated to assess the combined temperature and force influence and the temperature effect.