Nuclear Engineering and Design 246 (2012) 198–202

Contents lists available at SciVerse ScienceDirect

Nuclear Engineering and Design

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

# Mechanical properties and microstructural characterizations of potassium doped tungsten

Hua Sheng[a,b,*], Inge Uytdenhouwen[a], Guido Van Oost[b], Jozef Vleugels[c]

[a] SCK•CEN, The Belgian Nuclear Research Centre, 2400 Mol, Belgium
[b] Department of Applied Physics, Ghent University, J.Plateaustraat 22, B-9000 Ghent, Belgium
[c] Department of Metallurgy and Materials Engineering, K.U.Leuven, Kasteelpark Arenberg 44, B-3001 Leuven, Belgium

ARTICLE INFO

*Article history:*
Received 9 April 2011
Received in revised form
22 September 2011
Accepted 4 October 2011

ABSTRACT

Tungsten is a very promising candidate material for plasma facing components in fusion reactor due to its high melting temperature, high thermal conductivity, low tritium inventory and low erosion rate under plasma loading (Mitteau et al., 2007). The main drawback is the embrittlement at low temperature. The potassium doped tungsten grade WVWM produced by Plansee AG could be a potential plasma facing material for future nuclear fusion facilities and reactors such as ITER and especially DEMO.

For a better understanding of both recrystallization and ductile to brittle transition temperature, tensile tests are performed on potassium doped tungsten (WVWM), up to 2000 °C at different loading rates (0.2 and 42 mm/min). The mechanical properties are highly dependent on the microstructure. The fracture surfaces after tensile testing are microstructurally assessed by scanning electron microscopy (SEM) and transmission electron microscopy (TEM) is used to investigate the original specimen as the beginning point.



## 1. Introduction

Tungsten and tungsten alloys are presently considered for the helium cooled divertor and possibly for the protection of the helium cooled first wall in DEMO designs. Mainly because of their high temperature strength, good thermal conductivity and low sputter rates. The material has also to be stable under high neutron irradiation and helium and hydrogen production rates (Norajitra et al., 2010). None of the W & W alloys developed so-far has been fully optimized for structure or armour application in fusion reactors. In addition, all the present grades exhibit low fracture toughness and high ductile-brittle transition temperature (DBTT) in their initial metallurgical condition and are further degraded after neutron irradiation. From a material science point of view, the DBTT is not a real material property. It depends on the stress state (tensile, compressive or shear stresses), the strain state (uniaxial, biaxial or triaxial) and the strain rate (Hirai et al., 2007). Comparison between different grades is possible only if similar conditions are used. However, DBTT cannot be directly transferred to design specifications. As a conclusion, fracture toughness versus temperature is much better and more useful for design purposes. No full characterization has been performed up to now for a reference tungsten grade that can be used for comparison (Rieth et al., 2010).

The potassium-doped tungsten grade “WVWM”, is fabricated by the company PLANSEE AG. The purpose of introducing potassium into tungsten is to create bubbles in the material which pin the grain boundaries. Because potassium is volatile above 740 °C, different methods to keep potassium inside the material is used until there are no more open pores through which the potassium could leave. The growth of these bubbles due to induced stress by creep or others (potassium diffusion assisted) could be detrimental and is particularly seen for small wires in light bulbs. For bulk materials, it does not seem to be a critical issue. Through the addition of potassium to tungsten and the use of pressing, sintering and thermomechanical treatment, a fibrous elongated structure can be produced which combines high temperature creep resistance with a low temperature ductility and a better corrosion resistance. Hence this paper focuses on (1) understanding the mechanical properties of potassium doped tungsten in both as-received and annealed state; (2) studying the stress–strain relationships at 5 different temperatures up to 2000 °C by high temperature tensile testing; (3) investigating

* Corresponding author at: SCK•CEN, The Belgian Nuclear Research Centre, 2400 Mol, Belgium. Tel.: +32 14 332250; fax: +32 14 321216.
*E-mail addresses:* hua.sheng@sckcen.be (H. Sheng), iuytdenh@sckcen.be (I. Uytdenhouwen), guido.vanoost@ugent.be (G. Van Oost), Jozef.vleugels@mtm.kuleuven.be (J. Vleugels).

0029-5493/$ – see front matter 
doi:10.1016/j.nucengdes.2011.10.008

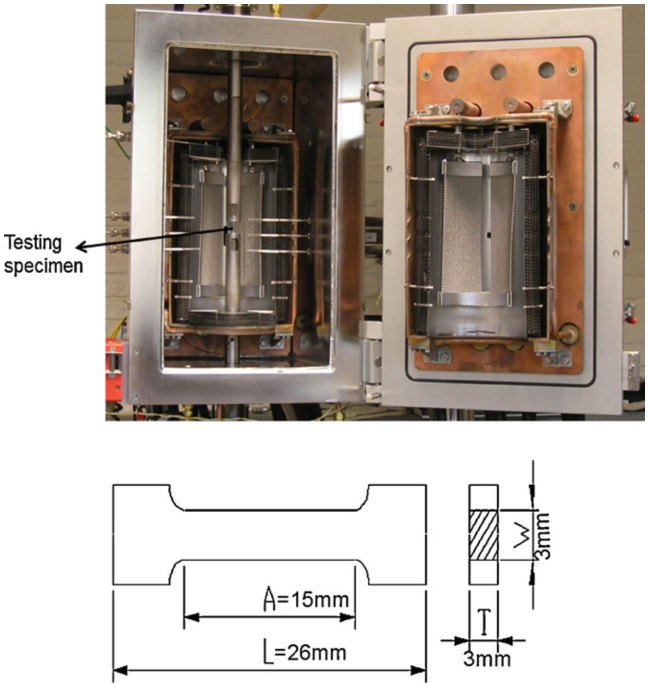


**Fig. 1.** *Top*: Digital picture of the inside view of the high temperature vacuum furnace for mechanical tests up to 2000 °C. *Bottom*: Drawing of a typical tensile specimen with dimensions.

microstructure of this WVWM by SEM and TEM in order to obtain better understanding of the material's real nature.

## 2. Experimental work

### 2.1. Material

The potassium doped tungsten (WVWM) is delivered in the form of forged rods with a diameter of 15 mm and a deformation degree of 1.7 (Pintsuk and Uytdenhouwen, 2010). The purity of this tungsten is above 99.99%, only 20–30 ppm of potassium is introduced by creating bubbles in tungsten which pin the grain boundaries.

### 2.2. Equipment

#### 2.2.1. High temperature tensile testing machine

Tensile testing was performed in the high temperature vacuum furnace shown in Fig. 1. Actually the tensile testing is performed on a mechanical test bench and the heating is done simultaneously on the specimen in the furnace. The tensile test specimens are dog-bone shaped with an overall length of 26 mm, a gauge length of 15 mm and an effective cross section of 3 mm × 3 mm as shown in Fig. 2. The tests were conducted in air at 300 °C, 500 °C, and in vacuum of $10^{-2}$–$10^{-3}$ mbar at 1000 °C, 1500 °C and 2000 °C (Uytdenhouwen, 2011). The temperatures were achieved by heating and cooling rates of 30 °C/min and using a 10 kN load cell. For the alignment of the specimen a preloading of 150 N, which corresponds to an initial stress of 17 MPa, was applied. The behaviour of BCC metals shows a strong dependence of the yield stress on the strain rate and temperature. Moreover, the yield stress of tungsten is strongly temperature dependent, and one also expects a strong rate dependence of the flow stress (Rieck, 1967). Therefore, the tests were performed with strain control and two different deformation speeds were applied, i.e. 0.2 mm/min on as-received and annealed (heat treatment at 1800 °C for 2 h in vacuum) WVWM and 42 mm/min only on the annealed material.

#### 2.2.2. Scanning electron microscopy (SEM)

To investigate the fracture surface in higher magnification, a scanning electron microscope (JXA 840 by JEOL) was used. All of the images in this paper were obtained from the secondary electrons.

#### 2.2.3. Transmission electron microscopy (TEM)

The specimens were investigated in a JEOL 3010 TEM operating at an accelerating voltage of 300 kV. Conventional bright field, dark field and electron diffraction techniques were used.

## 3. Result and discussion

### 3.1. The influence of strain rate on the ductility and the strength of annealed potassium doped W

The stress–strain curves for increasing temperatures for the as-received material tested at low deformation speed are shown in Fig. 2A. A large deflection in the slope for temperature at and above 1000 °C imposes the contribution of creep. At 300 °C, a large scatter in the stress–strain curves was found because it lies in the DBTT range of the material. Some of them fail prematurely while others exhibit already a non-negligible amount of ductility (up to 20%). Compare to it, the stress–strain curves with the same deformation speed after annealing behave entirely different (see Fig. 2B). The scatter in the data is rather acceptable. The curves are very reproducible both in the shape of the curve and ductility. In Fig. 2C, one can see that rather similar shaped stress–strain curves were obtained for the high deformation speed of 42 mm/min. The main difference at first sight from the curves is the higher brittleness at low temperature of 300 °C and increased ductility at 2000 °C (Uytdenhouwen, 2011).

### 3.2. Strength versus temperature and their dependence on the microstructure

A comparison of the yield strength (YS) and ultimate tensile strength (TS) from the as-received and annealed material is shown in Fig. 3 together with the strain. The first and most obvious result of the tensile test at 0.2 mm/min is that after annealing a drop of tensile strength by around 100 MPa compared to the as-received material is observed at all temperatures up to 1500 °C (Fig. 3 left). Furthermore it is accompanied by a significant increase in materials ductility (Fig. 3 right). At 2000 °C, which is above the recrystallization temperature, identical values were found for both materials owing to the recrystallization of the as-received material during test. This is rather an obvious result of the fact that the annealed material was heat treated at 1800 °C.

The above mentioned increase in ductility is of particular interest at 300 °C providing fracture strains of more than 30% for the recrystallized material while in its initial state the material still fails partly by premature fracture indicating that it is still not above DBTT. This can also be observed from the microstructure aspect. Fig. 4A–C shows SEM images of the fracture surface of the annealed and as-received WVWM tested at 300 °C. The as-received material (Fig. 4A) fractured 100% brittle with a typical cleavage pattern. The annealed material (Fig. 4B and C) only fractured partly brittle.

Microstructural investigations were also performed and the recrystallization temperature as well as related material modifications determined. A transition from transgranular cleavage at 300 °C to intergranular fracture and finally pure ductile fracture at 1500 °C, with a typical character of dimples in Fig. 4J–L, was found. At 2000 °C, which is above the before determined recrystallization temperature, identical values were found for both materials owing to the recrystallization of the as received material during the test. Meantime, the grain growth is obvious see in Fig. 4M–O. For the

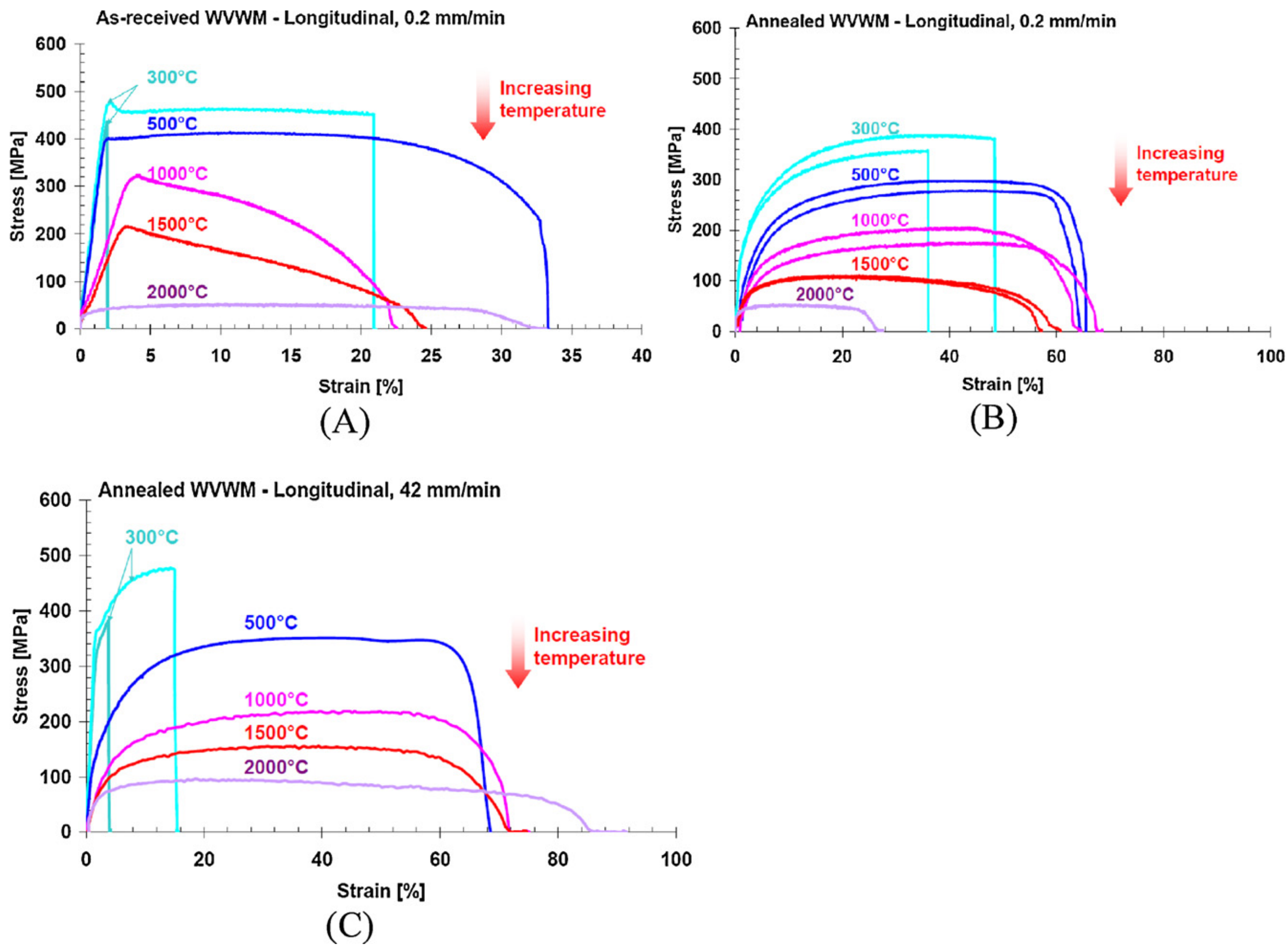


**Fig. 2.** Stress–strain curves for different temperatures at two different deformation speeds for both as-received and annealed WVWM. (A) As-received at low deformation speed (0.2 mm/min), (B) annealed at low deformation speed (0.2 mm/min), and (C) annealed at high deformation speed (42 mm/min).

annealing specimen, 3 stages were experiences, first is the recovery phase, which results in softening of the metal through removal of crystal defects (the primary type of which is the linear defect named a dislocation) and the internal stresses which they cause. It is well proved by TEM investigation. Recovery phase covers all annealing phenomena that occur before the appearance of new strain-free grains. The second phase is recrystallization, where new strain-free grains nucleate and grow to replace those deformed by internal stresses. If annealing is allowed to continue once recrystallization has been completed, grain growth will occur, in which the microstructure starts to coarsen. The latter two phases were observed by SEM.

### 3.3. Preliminary results of TEM investigation on WVWM

Even in chemically pure tungsten a perfect crystal lattice will never occur. The number and type of defects largely depend on the temperature treatment and deformation of the material (Rieck, 1967). Due to the limited amount of potassium, only 20–30 ppm, it could not be observed by TEM analysis. In Fig. 5, one can see quite

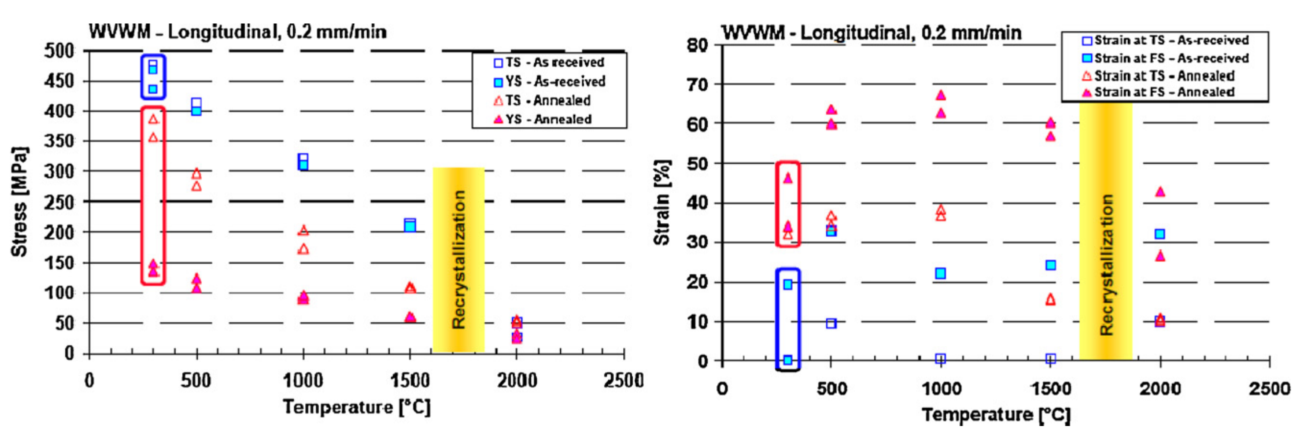


**Fig. 3.** Comparison of as-received and annealed WVWM at a deformation speed of 0.2 mm/min as a function of test temperature. *Left*: yield and tensile strength, *right*: strain at tensile strength and fracture.

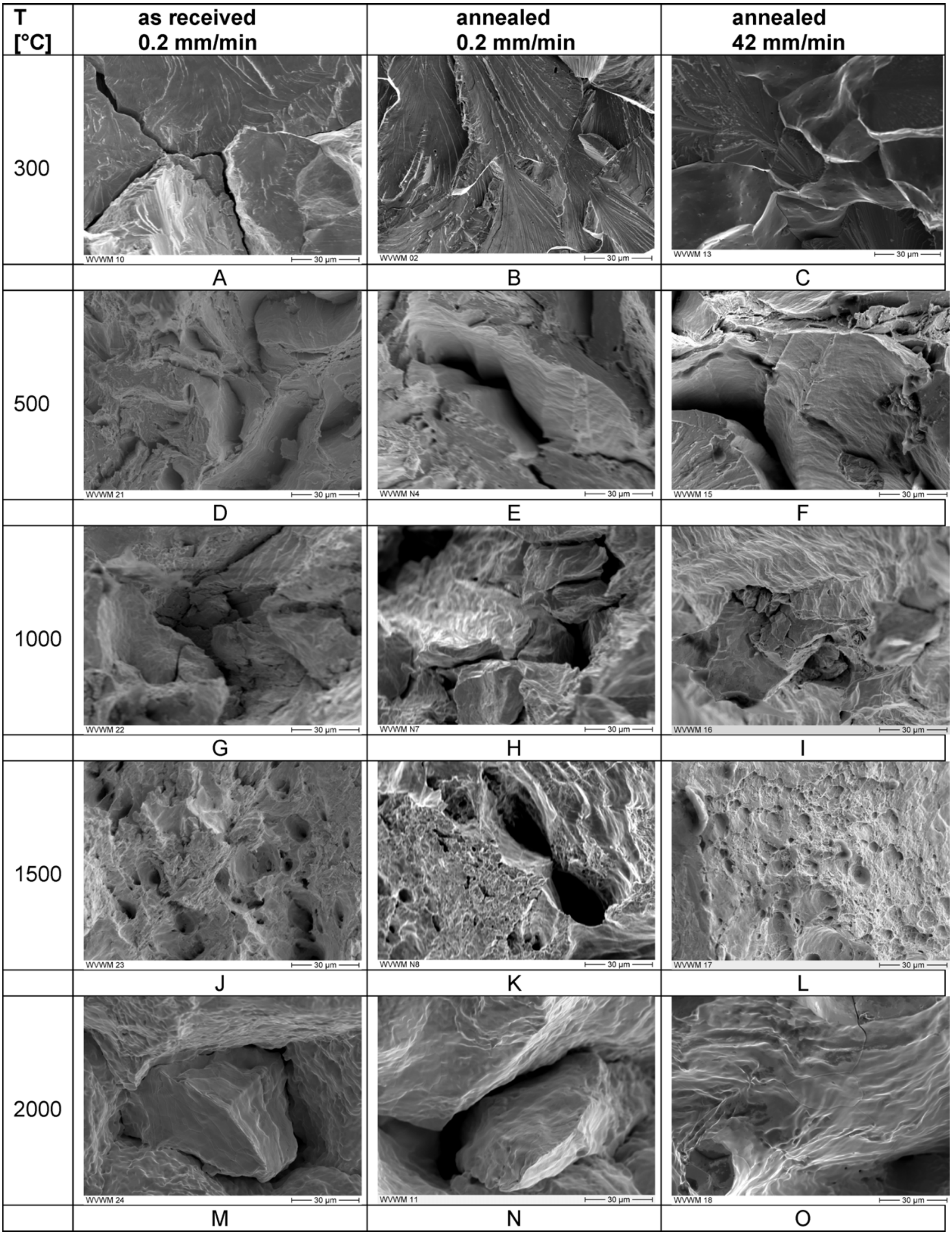


**Fig. 4.** Overview of SEM images of the fracture surfaces of WVWM after tensile testing, both as-received and annealed at two deformation speeds of 0.2 mm/min and 42 mm/min.

few short dislocation lines in both bright field image (indicated by blue arrows) and dark field image (obvious seen on the left and bottom of the image) of as-received WVWM.

Compared to Fig. 5, there is no visible dislocation lines can be seen in Fig. 6, which are the bright and dark field images of an annealed WVWM specimen. This can be explained by the nature of annealing. Annealing occurs by the diffusion of atoms within a solid material, so that the material progresses towards its equilibrium state. Heat is needed to increase the rate of diffusion by providing the energy needed to break bonds. The movement of atoms has the effect of redistributing and destroying the dislocations in the as-received WVWM. This alteration in dislocations allows metals to

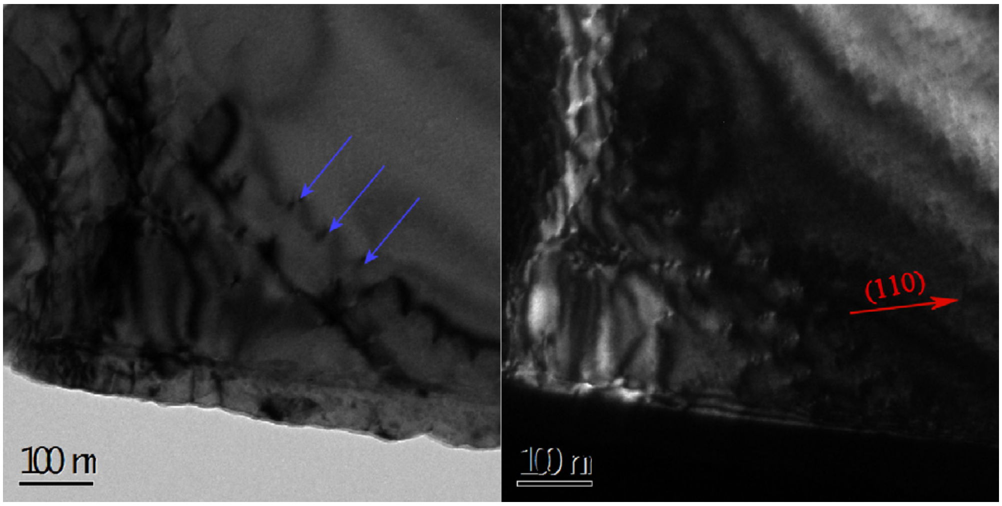


**Fig. 5.** TEM image of as-received WVWM. *Left*: Bright field image, *right*: dark field image with the diffraction vector of (1 1 0).

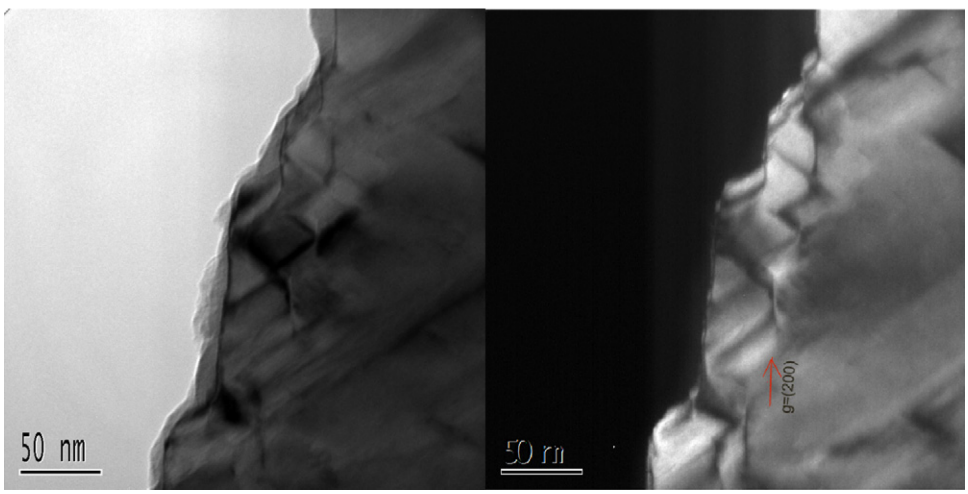


**Fig. 6.** TEM image of annealed WVWM. *Left*: Bright field image, *right*: dark field image with a diffraction vector of (2 0 0).

deform more easily, so increases their ductility as was also found in the mechanical properties.

## 4. Conclusions

To summarize, potassium doped tungsten is promising as one of the candidates in fusion environment. The mechanical properties from the annealed material showed acceptable behaviour especially with respect to the ductility. The decrease in strain rate improved the ductility even further. Furthermore, all the data obtained from mechanical testing have been strongly supported by their microstructure images as evidence.


## Acknowledgments

The authors greatly appreciate the scholarship awarded to Hua Sheng and financial support from SCK•CEN (Belgium).


## References


Hirai, T., Maier, H., et al., 2007. R&D on full tungsten divertor and beryllium wall for JET ITER-like wall project. Fusion Engineering and Design 82 (15–24), 1839–1845.

Mitteau, R., Missiaen, J.M., et al., 2007. Recent developments toward the use of tungsten as armour material in plasma facing components. Fusion Engineering and Design 82 (15–24), 1700–1705.

Norajitra, P., et al., 2010. He-cooled divertor for DEMO: status of development and HHF tests. Fusion Engineering and Design. 85 (10–12), 2251–2256.

Pintsuk, G., Uytdenhouwen, I., 2010. Thermo-mechanical and thermal shock characterization of potassium doped tungsten. International Journal of Refractory Metals and Hard Materials 28 (6), 661–668.

Rieck, G.D., 1967. Tungsten and Its Compounds. Pregamon Press Ltd., Headington Hill Hall, Oxford.

Rieth, M., et al., 2010. Review on the EFDA programme on tungsten materials technology and science. Journal of Nuclear Materials 417 (1–3), 463–467.

Uytdenhouwen. I., Degradation of first wall materials under ITER relevant loading conditions, ISBN 978-90-8578-406-7. Ph.D. Thesis, 2011.