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## High temperature fracture experiments on tungsten-rhenium alloys ☆

Stefan Wurster a,\*, Bernd Gludovatz a,b, Reinhard Pippan a,b

- <sup>a</sup> Erich Schmid Institute of Materials Science of the Austrian Academy of Sciences, Jahnstrabe 12, A-8700 Leoben, Austria
- <sup>b</sup> CD Laboratory of Local Analysis of Deformation and Fracture, Jahnstrape 12, A-8700 Leoben, Austria

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#### ABSTRACT

A big problem when using tungsten as plasma facing components in a future fusion reactor is the very low fracture toughness at low temperatures. Tungsten-rhenium alloys outclasses other tungsten-based materials in terms of increased ductility. We study the reason for this positive effect by investigating the influence of rhenium on the fracture process of tungsten-rhenium alloys at different temperatures (between room temperature and 900 C). The experiments are performed in a furnace-equipped tensile testing machine with a vacuum chamber, which allows us to perform fracture experiments at elevated temperature without oxidizing the material. Antecedent and subsequent electron backscattered diffraction scans are used to analyse the extent of plastic deformation and the interaction of plastic deformation and the fracture process. Furthermore, the consequences of recrystallization on the fracture process of tungsten-rhenium alloys will be analysed.

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#### 1. Introduction

Tungsten materials are considered to be implemented in the International Thermonuclear Experimental Reactor (ITER) in Cadarache, France, and in future fusion reactors as plasma facing materials in the main chamber, other proposed materials are beryllium and carbon fibre composites. Tungsten-based components, either in the shape of bulk or coated parts, will be placed in the divertor region [1]. The first wall, enclosing the plasma, is exposed to challenging conditions: high thermal fluxes up to several MWm<sup>-2</sup>,high operational temperatures and large temperature gradients. Exposure to radiation has to be considered too. Bolt et al. [2] estimate the neutron flux at the first wall of first commercial fusion power reactors after ITER to lead to 150 displacements per atoms (dpa). Beside good mechanical properties at high temperatures, tungsten and tungsten alloys feature high melting points and other superior thermal properties such as good thermal shock resistance and good thermal conductivity. In comparison to low-Z materials like beryllium and carbon, tungsten has a lower erosion rate. A disadvantage of high-Z materials in general is the lower tolerable impurity concentration inside the fusion reactor's plasma; otherwise, the radiation losses of the plasma would be too high. This fact might be outbalanced by the lower sputtering

E-mail address: stefan.wurster@unileoben.ac.at (S. Wurster).

yield of tungsten. Another disadvantage of tungsten in particular is its low fracture toughness, low elongation and small reduction in area at fracture at low temperatures, respectively its high ductile to brittle transition temperature (DBTT), complicating its machinability at room temperature. Nevertheless, the capability of application of tungsten as a plasma facing material has already been proven in the ASDEX (Axially Symmetric Divertor EXperiment) Upgrade tokamak [2].

It has been well known since the sixties and seventies, after intens research programmes related to aircraft and spacecraft technique, that alloying of tungsten with rhenium improves the mechanical behaviour of tungsten, which means lowering of DBTT [3-6]. In the following decades, the information on e.g. fracture behaviour of W-Re alloys is rather sparse [7], which might be related to the fact that Re is a very scarce - its abundance is more likely to be measured in ppt instead of ppm - and thus expensive metal. It is not planned to be used at high concentrations at a large, industrial scale. Nevertheless, not negligible amounts of rhenium and in succession osmium will be produced by transmutation of W due to neutron irradiation [8], implicating the existence of Re within the fusion reactor even when assuming pure W to be the used. However, the development of a strategy to enhance the mechanical behaviour of tungsten alloys is the main reason why the investigation is of interest.

#### 2. Experimental procedure

For fracture toughness experiments, we used an alloy of nominally 26% Re. Closer examination of the as-received material

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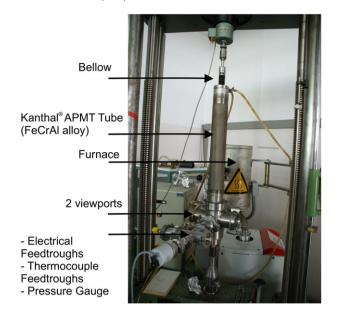
<sup>\*</sup> Corresponding author: S. Wurster, Erich Schmid Institute of Materials Science, Jahnstrasse 12, A-8700 Leoben, Austria. Tel.: +43(0)3842804325; fax: +43(0)3842804116.

with energy-dispersive X-ray spectroscopy (EDX) determined the rhenium content to be 26.8 wt.% corresponding to 26.5 at.%. The small difference in atom- and weight-percent is related to the close proximity of tungsten (No. 74) and rhenium (No. 75) in the periodic system of elements. No evidence was found that rhenium and tungsten are no solid solution, although the composition of the presented alloy is quite close to the brittle -phase. Out of an asforged rod with a diameter of 18 mm, compact tension (CT) specimens with thicknesses of *B* 3 mm and widths *W* 6 mm were manufactured.

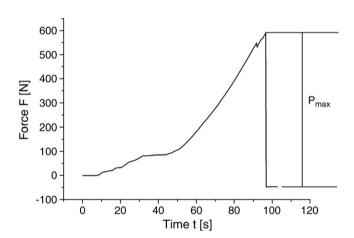
In this study, we compare the fracture behaviour of the asworked and subsequently stress-relieved W26Re alloy with the recrystallized alloy. After recrystallization of the specimens for 2 h at 2000 C in hydrogen atmosphere, notches (length 3 mm) were produced with a cutting disc in C-R-direction according to ASTM E399-90 [9]. The first letter of this code – used for describing crack geometry in relation to the rod axis - represents the direction normal to the crack plane and the second letter represents the direction of expected crack propagation. Assuming a cylinder, representing the starting material, circumferential (C), longitudinal (L) and radial (R) directions form an orthogonal coordinate system, in which the crack system is positioned. After recrystallization, the previously chosen crack orientation should not have a significant influence on the fracture behaviour due to the more globular shape of the grains and low crystallographic texture. The blunt notches were sharpened by succeeding razor blade polishing technique and cyclic compression [10] up to several 10,000 cycles; the result was a sharp pre-crack penetrating the whole width of the specimen. The influence of recrystallization on the microstructure is visible when checking Fig. 3(a) against Fig. 3(b) and (c). The well recrystallized microstructure is clearly evident from the uniform colour of the grains. Forging induces significant changes crystal orientation in the grains and the formation of a subgrain structure becomes visible in Fig. 3(a).

In addition to the standard microstructural characterization, electron backscatter diffraction scans (EBSD) were made before and after recrystallization of the fracture mechanics samples by use of a Zeiss 1525 scanning electron microscope equipped with an EDAX EBSD system. Evaluation of scans was made with Orientation Imaging Microscopy (OIM) software. Using the support of image quality maps, provided by OIM software, the ends of cyclic-compression-induced cracks are easily allocatable. Scans made after recrystallization and before breaking the specimens can then be compared with scans made after the experiment (Fig. 3(c)). This picture permits to estimate the amount of plastic deformation and the size of the plastically deformed region at different temperatures.

Specimens were tested in atmosphere at room temperature and at 300 C and in a furnace- and vacuum chamber equipped tensile testing machine (Fig. 1) at higher temperatures. The vacuum chamber is capable of reaching pressures of 2  $10^{-5}$  mbar. During the experiments, when reaching temperatures up to about 900 C,



**Fig. 1.** Assembly of the cylindrical vacuum chamber mounted inside the furnace-equipped tensile testing machine. The device is also equipped with a potential drop method measurement to determine the crack extension during loading.



**Fig. 2.** Load vs. time recording for specimen tested in atmosphere at 300 C,  $P_{\rm max}$  is taken for evaluation of  $K_{\rm O}$ .

the crosshead are recorded. The crosshead speed was set at  $0.4 \,\mathrm{mm \; min^{-1}}$  for all experiments. An example for recorded load vs. time diagram is shown in Fig. 2, for a specimen tested at 300 C in atmosphere. As it is shown there, the maximal difference in force, representing  $P_{\mathrm{max}}$ , is taken for evaluation of a conditional  $K_{\mathrm{Q}}$  value for fracture toughness according to following equation [9]

$$K_{Q} = \frac{P_{Q}}{B * W^{1/2}} * \frac{\left(2 + \frac{a}{W}\right) * \left(0.886 + 4.64 * \frac{a}{W} - 13.32 * \frac{a^{2}}{W^{2}} + 14.72 * \frac{a^{3}}{W^{3}} - 5.6 * \frac{a^{4}}{W^{4}}\right)}{\left(1 - \frac{a}{W}\right)^{3/2}}$$
(1)

the pressure increases but does not exceed  $10^{-2}$  mbar.  $1000 \, \text{C}$  is the as-designed temperature limit of the furnace. Temperature is measured with thermocouples of type K. Force, temperature, time and – owing to a constant crosshead speed – the displacement of

This test value,  $K_Q$ , as well as the experiment itself has to fulfill certain requirements to represent a valid  $K_{\rm IC}$  value. The size of the specimen has to fulfill: B and a > 2.5 ( $K_{\rm IC}/\gamma_{\rm S}$ )<sup>2</sup>.  $\gamma_{\rm YS}$  is the 0.2% offset yield strength of the material for the temperature and loading rate

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