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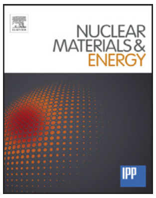

Improved fracture behavior and microstructural characterization of thin tungsten foils

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ABSTRACT

This study is focused towards the development of the technique for investigating the fracture behaviour of 100 µm thick rolled tungsten foils, with a purity of 99.97%. Electron backscatter diffraction (EBSD) scans reveal that the grains are elongated along the rolling direction of the foil, which has a very strong $\{100\}<011>$ texture. The test specimens were fabricated by electrical discharge machining (EDM) and cracks were initiated by consecutively using a diamond wire saw, a razor blade and a focused ion beam (FIB) workstation. Fracture experiments were performed at temperatures from −196 °C to 800 °C. The investigation of fracture appearance shows an improved behavior and significantly higher values of conditional fracture toughness K_q compared to bulk W-materials, which can be related to a higher degree of deformation during the production process. A high toughness at room temperature (RT) and 200 °C, slowly decreases when approaching the highest testing temperature of 800 °C. The most significant result reveals that the ductile to brittle transition temperature (DBTT) is around RT, which is an extraordinary result for any tungsten material. The fracture surfaces, investigated with a scanning electron microscope (SEM), show a transition from cleavage fracture at liquid nitrogen temperature, through pronounced delamination within the foil plane at ambient temperatures to ductile fracture at the highest testing temperatures.

1. Introduction

Tungsten (W) is a metal with an excellent combination of high temperature properties, such as high erosion resistance, low vapor pressure and high strength at elevated temperatures. It also has the highest melting point of all metals ($T_m = 3422$ °C) and other superior thermal properties: good thermal shock resistance and high thermal conductivity. These exceptional qualities qualify this metal for many high temperature applications, like e.g. in a future fusion reactor. Extensive research on divertor design concepts is directed towards the use of various tungsten materials and its composites for different divertor applications, making tungsten one of the main armour and heat sink materials candidates [1,2]. A divertor is one of the key in – vessel components of a fusion reactor, responsible for power exhaust and cleaning the plasma from He and impurities i.e. various particles coming from the first wall. Therefore, these reactor parts will be subjected to a very high heat flux loads: in a normal scenario 10 MW/m^2 is expected, having peaks of up to 20 MW/m^2 in off normal events, as a consequence of different plasma instabilities [3,4]. For such a challenging and extreme thermal application, tungsten was a natural choice as a candidate material.

When thinking of using tungsten as a structural part for the divertor region, its main disadvantage – inherent brittleness - plays a decisive role. Tungsten shows low fracture toughness at low temperature and like other body centered cubic (bcc) metals, it has a transition from brittle fracture at low temperatures to ductile and tough behavior at high temperatures. The problem is that the transition temperature is relatively high (well above ambient temperatures), additionally complicating the machining at room temperature (RT). Several ductilization strategies have been proposed in the attempt of overcoming the main problem of tungsten and improving its fracture behavior, including toughening tungsten by synthesis of i) solid solutions, ii) nanostructured materials and iii) various tungsten composites. Tungsten laminates synthesized of ultra – fine grained tungsten foil [5,6] is an approach towards making tungsten – based materials more ductile and expanding its application from armour to structural materials. Some divertor

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concepts being currently investigated might use He as a coolant and would require the use of W in form of cooling pipes, which will be exposed to 600 °C and 100 bar [7]. Typical manufacturing processes like extrusion or drilling of holes in rods are challenging and additionally, in this way, the alignment of elongated grains i.e. the preferred crack propagation path would be coinciding with the expected fracture direction of the pressurized pipe. Therefore, the option of synthesizing the structural tungsten pipe by rolling up and joining of tungsten foils seems to be very promising, since the best case scenario of microstructural design, where the direction of low fracture toughness follows the contour of the pipe, can be obtained. The 100 µm thick foil used for forming of pipes shows some extraordinary mechanical properties, extending the foil's RT ductility to a ductile bulk material [8].

The fracture behavior of tungsten materials has been studied for decades, but full understanding of underlying fracture processes, greatly influenced by different parameters, such as microstructure, has not been completely realized. Extensive studies of the fracture resistance of tungsten single crystals were performed in the nineties [9,10], revealing that {100} planes are primary cleavage planes with < 110 > being the preferred crack propagation directions. In addition, the strong loading rate dependence of the ductile to brittle transition temperature (DBTT), in particular at elevated temperatures, confirms that the transition is controlled by dislocation mobility [9]. In contrast to the good descriptions of fracture processes in single crystals, the comprehensive experimental study of tungsten materials with more complex microstructures i.e. industrially produced polycrystalline W is still not complete. However, this could be essential in order to identify ways of improvement and to develop microstructural design concepts with optimized fracture properties.

In the case of tungsten, factors like microstructure, grain size, grain shape, texture, etc. greatly influence the resulting mechanical properties. It has been shown that there is a strong correlation between manufacturing history (sintering, rolling, swaging, hot/cold work…) and the resulting material's microstructure and mechanical properties [11]. Among various parameters affecting the fracture toughness, the established microstructure during production steps plays a decisive role for the resulting failure mode, leading to the anisotropic behavior of deformed (worked) tungsten [12,13]. A pancake – like microstructure of tungsten plate materials is favorable for good toughness in two out of three possible testing directions and when decreasing the thickness of the material, a shift of DBTT to lower temperatures is also observed [14]. Furthermore, it was shown that low temperature rolling of commercially pure tungsten enhances its strength and ductility [15]. This leads to the conclusion that a thinner plate, which experienced a higher degree of cold work has a more beneficial microstructure, having smaller grains and higher amount of mobile edge dislocations.

So questions that need to be answered are: "How do fracture properties change if tungsten plate material is submitted to even higher degrees of rolling (deformation) resulting in commercially available 100 µm thin tungsten foil? Are the values of fracture toughness higher in comparison to other tungsten materials? What happens to the DBTT in case of such highly deformed foils?" It has already been shown that such a material behaves ductile in a tensile experiment at RT and when synthesized in laminated plates and tested with Charpy impact test, the DBTT can be reduced by 300 °C [8]. As mentioned in previous paragraphs, in some He – cooled divertor concepts the use of structural, pressurized W cooling pipe is being investigated where a ductile laminated pipe can be made of 100 µm ultra – fine grained W foil [7]. Therefore, from a materials design aspect, it is extremely important to determine fracture toughness values and investigate the effect of various testing conditions on the variation of fracture behavior of such a tungsten foil used as a base material in laminated tungsten composites. The aim of this work was to develop a testing procedure for evaluating fracture toughness on a 100 µm thin tungsten foil, investigating how variation of testing temperature affects the resulting fracture process.

2. Materials and microstructure

The fracture mechanical testing was performed on a 100 µm thin unalloyed tungsten foil, a standard material that is commercially available and produced by Plansee SE, Reutte, Austria. The tungsten plate is manufactured via the powder metallurgical route and after sintering, it is submitted to hot and cold rolling processes. As a result, a thin foil is obtained having elongated grains along the rolling direction. The dimension of grains in foil thickness direction is in ultra – fine grained regime having average grain diameter of less than half of micron. Production details regarding degree of deformation, temperatures of hot and cold rolling and stress relieve annealing conditions are not provided by the manufacturer. The guaranteed purity of the material is 99,97% and impurities content information can be found elsewhere [16].

In order to evaluate the microstructure and texture of the foil, a scanning electron microscope (SEM) (Leo 1525, Zeiss) with an electron backscatter diffraction (EBSD) detector was used. During preparation of the tungsten foil for EBSD analysis by embedding in hard compound and mechanical grinding and polishing, delamination along the grain boundaries occurs. To prevent this, ion polishing (Hitachi E-3500 Cross Section Polisher) of the desired cross section was performed. Resulting smooth surfaces across the entire thickness of the sample were suitable for following SEM/EBSD analyses. Evaluation of the obtained scans was made with Orientation Imaging Microscopy (OIM) software.

EBSD orientation imaging map of a pure W foil in as – received condition can be seen in Fig. 1(a), with distinctive elongated grains going along the rolling direction (from left to right) forming a typical "pancake" like microstructure. Such an appearance is a direct consequence of a high degree of deformation during production steps. Average grain size given by the ASTM number, calculated by enforcing the average grain area, is 19,6. The aspect ratio, one of the measures of the grain shape, is defined as ratio of the length of the minor axis divided by the length of the major axis. In the case of as received materials, average aspect ratio of grains is 0,45. Microstructure of a W foil after being exposed to 1080°C for 12 minutes is shown in the scan in Fig. 1(b) and will be further discussed in Chapter 4.1. The results of the EBSD scans can be presented in Pole Figures (PF) shown in Fig. 1(c). It can be seen that a 0.1 mm tungsten foil has a very strong texture in {100} < 011 >, indicating that the preferred cleavage planes {100} form an angle of 45° with the rolling direction. This result corresponds to other investigations [17] and such a texture established, during cold work by grain rotation, defines a saturation condition, meaning it does not change by further deformation [18].

3. Experimental procedure

3.1. Sample preparation

In this study, the main goal was to determine fracture toughness of a 0.1 mm thin foil, which at the initial stage required designing and manufacturing a suitable experimental setup. Since the thickness of the foil and its geometry are not the standard one as e.g. given in ASTM E399 [19], optimization of sample preparation, crack initiation and brazing process had to be performed. The preparation procedure can be summed in the following three steps:

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