International Journal of Refractory Metals & Hard Materials 72 (2018) 97–103

Contents lists available at ScienceDirect

International Journal of Refractory Metals & Hard Materials

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

Recrystallization and composition dependent thermal fatigue response of different tungsten grades

G. Pintsuk[a,*], S. Antusch[b], T. Weingaertner[b], M. Wirtz[a]

[a] Forschungszentrum Jülich GmbH, Institute for Energy and Climate Research, Partner of the Trilateral Euregio Cluster (TEC), 52425 Jülich, Germany
[b] Karlsruhe Institute of Technology (KIT), Institute for Applied Materials, 76344 Eggenstein-Leopoldshafen, Germany

ARTICLE INFO

Keywords:
Tungsten
Recrystallization
Thermal fatigue
High heat flux

ABSTRACT

Industrial pure tungsten grades, manufactured by using a variety of manufactured techniques, are available worldwide in many different types of semifinished products, i.e. rods, wires, ribbons, and sheets. Thereby, the recrystallization temperature varies depending on the applied degree of deformation but also depending on the materials composition, i.e. the materials purity and in particular the level of certain impurities.

In order to compare different available industrial tungsten grades and a newly developed PIM-W grade, on the one hand recrystallization studies at three different temperatures from 1573 to 2073 K for 1 h were performed using microstructural analyses and Vickers hardness testing. On the other hand, the thermal shock induced low cycle thermal fatigue response of the material in its different recrystallization stages was done using high heat flux tests at 1273 K base temperature, applying 1000 shots with 1 ms and 0.38 GW/m^2 and post-mortem characterization, i.e. profilometry and metallography. The obtained results are related to the microstructural and mechanical features of the materials as well as the chemical composition of the individual tungsten grades.

1. Introduction

The use of tungsten as plasma facing material for future nuclear fusion applications is on the one hand related to its beneficial properties like high melting temperature, good thermal conductivity and low tritium inventory. On the other hand, its brittleness at low temperatures and its recrystallization and therefore softening at high temperatures puts high demands on the component design as well influences the allowed operational temperature limits. Besides, also the material itself, its related manufacturing technology and impurity concentration, is expected to play a decisive role as it influences the materials microstructure, its anisotropic material properties and also recrystallization.

For the next step fusion device ITER ("The Way" in Latin) actually being built in Cadarache, France, tungsten monoblock-shaped components and qualification mock-ups schematically shown in Fig. 1 have been produced in Europe and Japan for the use in the divertor of the device [1,2,3,4]. These mock-ups not only use different tungsten grades from European and Russian on the one hand and Japanese manufacturers on the other hand but are also produced using different manufacturing technologies, i.e. hot isostatic pressing (HIP) and hot radial pressing (HRP) in Europe and brazing in Japan. Thereby, the manufacturing technology has to guarantee a sufficient remaining strength of the CuCrZr-tube acting as structural material, which requires dedicated annealing and temperating treatments depending on the process temperature during joining.

The manufactured mock-ups have been tested in electron beam facilities in France and Russia up to 1000 cycles at 20 MW/m^2 with 10 s loading and 10 s dwell time showing a different surface response depending on the used electron beam (beam diameter: locally induced thermal shock loads during beam scanning) and also a varying response with respect to the formation of macro-cracks (Fig. 1) for European and Japanese manufacturers [4,3,5]. While macro-cracks were found for all European manufacturers and an increased number of cracks was found in case of the existence of electron beam induced surface cracking acting as potential crack initiation points (modelling by Li et al. [6]), no macro-crack formation was found for the Japanese mock-ups. The influencing factors are assumed to be the thermal and mechanical resistance of the individual tungsten grades and the manufacturing technology. However, also the design of the components and in particular the geometrical dimensions of the tungsten block seem to play a decisive role, e.g. no crack formation was found for previously investigated designs with 23 mm block width and 10 mm inner cooling tube diameter, while crack formation occurs frequently in case of the actual design with 28 mm width. This observation is in agreement with the findings from modelling studies on the influence of the component size [7].

* Corresponding author.
E-mail address: g.pintsuk@fz-juelich.de (G. Pintsuk).

https://doi.org/10.1016/j.ijrmhm.2017.11.039
Received 21 September 2017; Accepted 25 November 2017
Available online 14 December 2017
0263-4368/

Fig. 1. a) Schematic view of a tungsten monoblock mock-up consisting of a CuCrZr-tube with 12 mm inner diameter and a thickness of 1.5 mm, a 1 mm pure copper interlayer and surrounding tungsten blocks with 28 × 12 mm front surface and a varying height (plasma front thickness 5.5 to 8 mm); b) Top view of such a mock-up with 7 tungsten blocks after testing at 20 MW/m^2.

In any case, by comparison of the results from the two different electron beam facilities, the statistical appearance of macro-cracks is significantly increased as soon as shallow surface cracks, introduced by the electron beam, are present. As such shallow cracks may also appear during operation as a result of highly frequent Edge Localized Modes (ELMs) applying thermal shock loads in the range of up to the GW/m^2 range in non-mitigated condition for about 0.5 ms.

The investigations herein focus on the characterization of the different commercially available tungsten grades that have been used for the fabrication of the European and Japanese mock-ups in comparison with a tungsten grade manufactured via powder injection molding (PIM) providing an isotropic and recrystallized microstructure directly after the manufacturing process. Thereby, the thermal shock induced thermal fatigue performance of the materials is determined in dependence on the materials microstructure, not only with respect to the manufacturing technology but also to their recrystallization behavior. These investigations should provide an answer to the question on the influence of the material on the formation of macro-cracks in the actively cooled components and provide information about damage characteristics due to thermal shock loading that have to be taken into account for the component design.

2. Materials microstructural and mechanical characterization

2.1. Tungsten grades & recrystallization

In the frame of this work, four different tungsten products were investigated, i.e. powder injection molded pure W produced by KIT (PIM-W) [8–10], a forged bar material produced by PLANSEE AG (W-PL), and rolled plate materials from POLEMA JSC (W-PO) and A.L.M.T. Corp (W-AL).

The industrially produced bar and plate materials, all of them fulfilling the actual specifications for the use as plasma facing material in ITER and being used for the manufacturing of the before mentioned ITER mock-ups, are characterized by an anisotropic grain structure and related material properties due to the applied deformation processes for densification. In contrast, PIM-W, being produced not yet at the industrial but medium scale level, exhibits a fully isotropic grain structure in the recrystallized state due to high final sintering temperatures > 2473 K (Fig. 2, left column).

The materials were characterized by chemical analyses to determine the exact composition of the investigated batch and any potential influence that may contribute to observed differences in the material behavior. The analyses were done by ICP-OES and ICP-MS for most of the elements while ON were analyzed by hot gas extraction and CS with a CS-analyzer. The obtained results are provided in Table I and indicate that O, Cr, Fe, Ni, Cu and Mo are the main impurities still on a low ppm level. Thereby, W-AL exhibits the highest purity among all investigated materials.

From the materials, small thermo-shock specimens with the size of 10 × 10 × 4 mm were cut by EDM in such a way, that the deformation direction of the grains is perpendicular to the surface. This orientation represents the configuration, which will be finally used for the manufacturing of actively cooled plasma facing components. Subsequently, individual specimens were annealed in a tungsten vacuum furnace (10^{-4}–10^{-5} mbar) at 1573 K, 1773 K and 2073 K for 1 h in Al_2O_3 crucibles. The heating rate was 10 K/min and the cooling rate for 1573 K and 1773 K was 10 K/min for 20 min followed by adiabatic cooling. For annealing at 2073 K, in order to protect the furnace and the used crucibles, the cooling rate was 6.7 K/min till 1873 K, 10 K/min till 1673 K, 12 K/min till 1373 K and finally adiabatic cooling. This aims for the determination of the onset of recrystallization for the particular material and the influence of recrystallization on the mechanical and in particular thermal-shock induced thermal fatigue performance of the material. After recrystallization both the reference and the annealed specimens were polished on one front surface to a mirror finish with a roughness R_a of ~0.1 μm.

In Fig. 2 the different microstructures of the materials as a function of the annealing treatment are shown with the view on the surface plane that will be exposed to the high heat flux loads. As mentioned before, the grain size (Fig. 3) and shape varies strongly between the different materials in their reference state. Thereby, for PIM-W it has to be taken into account that due to the net-shape production method without any further post-treatment it is characterized by a dual microstructure (Fig. 4a). This microstructure consists of a surface near seam of about 0.5 mm thickness exhibiting grains with a size of up to several hundred micrometers (surface near, i.e. the first 100–150 μm the grain size is smaller as shown in Fig. 4b) and a bulk grain structure with an average grain diameter of ~40 μm. As the thermal shock testing is a surface-near process, the material characteristics provided in the following are limited to those representing the surface-near region.

While PIM-W shows large isotropic grains whose shape and average size does not vary as a function of the annealing temperature, W-PL is characterized in the reference state by significantly smaller grains with a needle like structure oriented perpendicular to the surface. Due to annealing the anisotropy of the grains vanishes, partially at 1573 K and fully at 1773 K, and the size of the recrystallized grains is about 30% larger than the one for PIM-W. W-PO and W-AL, both rolled materials with a plate like grain structure with one deformation direction in the surface plane and the other perpendicular to the surface, exhibit a significantly smaller grain size with an elongation ratio of 1:4 and while here the recrystallization seems to be finalized already after annealing at 1573 K (almost in case of W-AL), the grain sizes are about 50% below those for PIM-W.

In addition, Auger electron spectroscopy of the fracture surfaces of the different materials were performed to determine existing impurities within the grains and at the grain boundaries. It was shown that the investigated specimens from W-PO and W-AL, in contrast to W-PL (for PIM-W the results were not conclusive), exhibit a small amount of phosphorus at the intergranular surfaces. In all cases, no phosphorus was found on the cleavage planes. How these findings correlate with the recrystallization performance of the material will be discussed below.