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Effect of thermal heat loads on the microstructure of recrystallized double forged tungsten

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HIGHLIGHTS

- The effect of electron exposure on W was studied with SEM and TEM.
- A sample preparation method was developed for TEM investigation about 10 μm below the surface.
- The deformation of the material by the thermal heat load is described.
- The changes in microstructure just below the exposed surfaces are analyzed.

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ABSTRACT

Two recrystallized double forged W materials, one which was pre-heated up to 400 °C and exposed to 120 keV electrons during 100 pulses of 1 ms giving a total power of 1.26 GW/m^2 and one as reference which was only pre-heated to 400 °C, were investigated with SEM and TEM to reveal the effect of the exposure on the microstructure. The reference material revealed that the selected material contains only some large angle grain boundaries and occasionally a few isolated dislocations. The e-beam exposure of the material resulted only in a deformation of the matrix. The surface has roughened and series of parallel ridges were observed in the SEM images. However, no cracks or other signs of material rupture were found. On a microscopic level the deformation manifested as an increased number of tangled dislocations and the reappearance of small angle tilt boundaries.

1. Introduction

Tungsten is considered as a material for first wall components in future tokamaks. During the operation in a tokamak, the first wall components will be exposed to heat loads due to the interaction of the plasma with the material. One of the interactions are edge localized modes (ELMs) which occur with a high repetition rate of more than 1 Hz and during which a significant amount of energy is deposited in a short time (<1 ms). Due to these thermal heat loads, the microstructure of the materials will be affected and the surface of the components may be damaged [1]. To guarantee the integrity of the plasma facing material, the effect of these thermal heat loads on the microstructure of the material needs to be investigated and understood.

However, until now, very few studies were performed to determine the effect of the exposure of W to the plasma. In particular, the effect on the microstructure is not known. In this paper, a first experiment is described. A recrystallized tungsten material was exposed to electrons to reveal the effects of the thermal heat load only. The changes in microstructure are revealed by a combination of scanning electron microscopy (SEM) and transmission electron microscopy (TEM).

2. Experimental

The tungsten material selected for this investigation was a double forged and recrystallized pure tungsten produced by Plansee AG, Austria. After recrystallization at 1600 °C for 1 h almost all defects were removed from the material [2], which makes it more suitable for this experiment.

The sample is a tablet of 10 mm × 10 mm × 4 mm. It was pre-heated to 400 °C and on the top surface an area of 4 mm × 4 mm was exposed to the fast scanning e-beam JUDITH at FZJ to electrons of 120 keV in 100 pulses of 1 ms. Due to the high acceleration voltage the electrons penetrate around 7 μm deep into the material and cause volumetric loading instead of surface loading within this thin layer. The total absorbed power density, calculated using an

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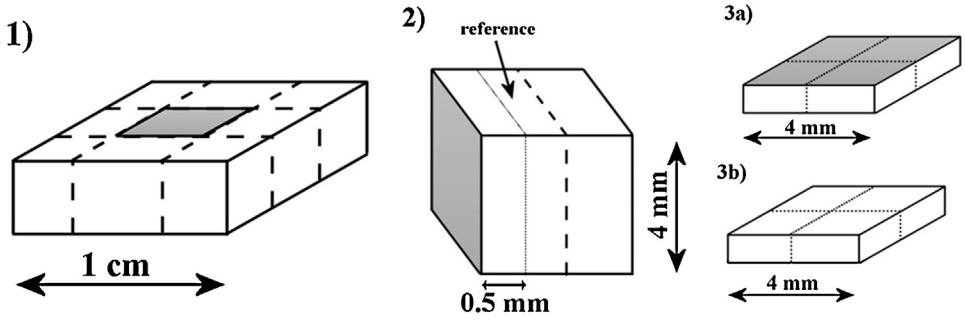

Fig. 1. Cutting scheme applied during sample preparation.

electron absorption coefficient of 0.46, equals to 1.26 GW/m^2. Even with this rather high applied loading, no melting was expected. Calculations predicted a maximum surface temperature of 2600 °C for a base temperature of 400 °C, which is well below the melting temperature of tungsten (3410 °C). Also no cracking was expected because previous studies showed that the cracking threshold for recrystallized double forged tungsten lies at base temperatures below 300 °C [3]. The penetration depth of the heat front during a 1 ms pulse was calculated to reach up to 245 μm, but the most affected areas are located closer to the exposed surface.

The sample preparation aimed at obtaining samples as close to the surface as possible. The cutting procedure is schematically represented in Fig. 1. First, the exposed area of 4 mm × 4 mm was cut out of the sample block. Then, two slices of about 0.5 mm thick were cut parallel to the exposed surface. The first slice contains the exposed material and the second slice is the reference material. Finally, both slices were cut in four to have samples that are smaller than a 3 mm disc.

These samples were mechanically polished on SiC paper with grit sizes of 500, 1200 and 4000. The reference material was polished on both sides. The exposed material was only polished from one side to avoid that the affected material is removed. It was verified that the final thicknesses of all specimens varied between 50 and 100 μm.

All specimens were glued on 3 mm copper grids with a circular aperture of 1 mm with the exposed surface facing the grid. The final step in the specimen preparation is electrochemical polishing at room temperature using an electrolyte consisting of 1.5 wt% NaOH in water and applying a voltage of 15 V. A combination of double- and single-jet polishing was applied. First the specimen was polished from both sides for 30 s. During this time, a thin layer of about 5–10 μm was removed, which was necessary to remove the surface roughness. In a second step, the exposed side was protected from the polishing liquid and material was removed from the non-exposed side until perforation.

The specimens were investigated with TEM on a JEOL 3010 microscope operating at 300 kV. A few SEM images, recorded on a JEOL 840 microscope, are discussed as well.

3. Results

3.1. SEM investigation

Fig. 2a shows a SEM image of the electron beam exposed surface before electrochemical polishing. A series of parallel ridges of a few μm high and spacing of 5–10 μm can be observed. The indicated ridge height is only an estimate because height differences cannot be measured accurately in this way. No surface roughness was observed at the tablet surface that was not exposed, indicating that the ridges are the result of the exposure.

Apart from the roughening of the surface, no cracking or other damage effects were found. During a typical thermal shock event, the huge temperature gradient creates large thermal stresses parallel to the surface. Since the sample was preheated up to 400 °C the material was deformed plastically. Therefore, stress release induced by the large temperature gradients occurred only by plastic deformation instead of crack formation. Fig. 2b shows the exposed specimen after electrochemical polishing. The double sided polishing for 30 s is sufficient to remove the roughened area. Based on the total polishing time and the initial specimen thickness, it is estimated that a layer of 5–10 μm is removed from the exposed surface, which is sufficiently close to provide relevant data.

The intensity differences in the detailed image of Fig. 2b are due to small angle and large angle grain boundaries. In the lower half of the hole, a thick line is intersecting the hole. The surface

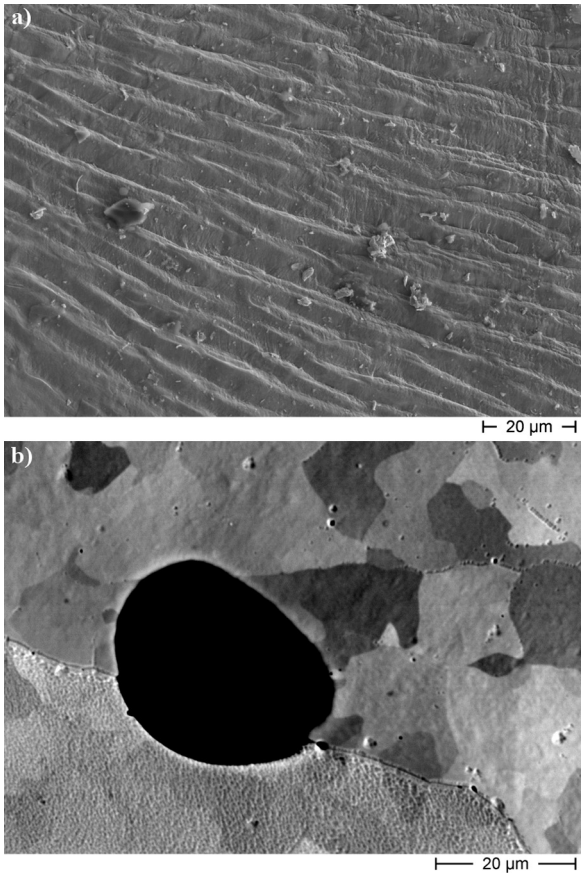

Fig. 2. SEM image of the exposed surface (a) before and (b) after electrochemical polishing.