Journal of Nuclear Materials 477 (2016) 77–84

Contents lists available at ScienceDirect

Journal of Nuclear Materials

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

Investigation of the combined effect of neutron irradiation and electron beam exposure on pure tungsten

W. Van Renterghem*, I. Uytdenhouwen

SCK·CEN, The Belgian Nuclear Research Centre, Boeretang 200, 2400 Mol, Belgium

HIGHLIGHTS

- Neutron irradiated and electron beam exposed tungsten samples were studied with transmission electron microscopy.
- Neutron irradiation creates dislocation loops and rafts, while voids are created at higher irradiation dose.
- No precipitates of transmutation products were found under these low dose irradiation conditions.
- Electron beam exposure annihilates the dislocation loops and rafts.

ARTICLE INFO

Article history:
Received 29 February 2016
Received in revised form
19 April 2016
Accepted 3 May 2016
Available online 7 May 2016

Keywords:
Tungsten
Neutron irradiation
Electron beam exposure
Transmission electron microscopy

ABSTRACT

Pure tungsten samples were neutron irradiated in the BR2 reactor of SCK·CEN to fluences of 1.47×10^{20} n/cm^2 and 4.74×10^{20} n/cm^2 at 300 °C under Helium atmosphere and exposed to the electron beam of the Judith 1 installation The effect of these treatments on the defect structure was studied with transmission electron microscopy. In the irradiated samples the defect structure in the bulk is compared to the structure at the surface. The neutron irradiation created a large amount of a/2⟨111⟩ type dislocation loops forming dislocation rafts. The loop density increased from 8.5×10^{21}/m^3 to 9×10^{22}/m^3 with increasing dose, while the loop size decreased from 5.2 nm to 3.5 nm. The electron beam exposure induced significant annealing of the defects and almost all of the dislocation loops were removed. The number of line dislocations in that area increased as a result of the thermal stresses from the thermal shock.

1. Introduction

The fusion of deuterium and tritium results in the formation of a helium atom and a neutron. These neutrons, which have an energy of 14.1 MeV, will interact with the reactor wall and affect the mechanical properties of the reactor materials. This interaction was not an issue in the existing fusion devices, because most experiments did not use the tritium gas. When it was used, the total amount of fusion reactions and hence the total amount of 14.1 MeV neutrons was small and the effect on the structural components was minimal. Therefore, only few studies on fusion materials focused on irradiation effects [1–3]. In future reactors, like ITER, DEMO, or eventually fusion power plants, the amount of neutrons will be considerably higher [4,5] and it must be studied which effect neutron irradiation will have on the mechanical properties of the materials that will be used. Tungsten is the main candidate for first wall and divertor materials [6,7] and will face the highest degree of neutron irradiation. Therefore, it's of primordial interest to study the effect of the neutron irradiation on the defect structure and structural integrity of tungsten materials.

A second reason why so few studies focus on irradiation effects in tungsten is that there is no installation available, at present, that produces large amounts of such high energy neutrons. Only nuclear fission test reactors are capable of producing high neutron fluxes, but the energy of these neutrons is at most a few MeV. Even though, exact irradiation conditions cannot be simulated, more and more studies appear on the effect of neutron irradiation of the behavior of materials [8–13]. Several other studies use self-ion irradiations [14–16].

Apart from the neutron irradiation, the first wall material will also be exposed to transient events of the plasma [17,18]. One of the events are disruptions during which a high amount of energy is

* Corresponding author. SCK·CEN, NMS-MNA, Boeretang 200, 2400 Mol, Belgium.
E-mail addresses: wvrenter@sckcen.be (W. Van Renterghem), iuytdenh@sckcen.be (I. Uytdenhouwen).

http://dx.doi.org/10.1016/j.jnucmat.2016.05.008
0022-3115/

transferred from the plasma to the reactor wall, giving a thermal load on the reactor wall material. These events do not occur very frequently and should be avoided as much as possible because the effect on the material integrity is very strong [18–20].

While a new irradiation facility that can perform irradiations with the same neutron energy spectrum as in a fusion reactor is still under construction [21], the study of the general effects of neutron irradiation and thermal loads on the candidate first wall materials is quite useful for their qualification as a fusion material. The experimental setup that most closely simulates both effects and that is available at this moment is the irradiation in a nuclear fission based material test reactor followed by electron beam exposure. Even though the neutron energy spectrum of a fission reactor is not the same, the qualitative aspects of neutron irradiation can be studied. Electron beam test facilities, for their side, are routinely used to investigate the effect of thermal loads experimentally [22].

For this study, tungsten was irradiated in the BR2 material test reactor of SCK·CEN to fluences of 1.47×10^{20} n/cm^2 and 4.74×10^{20} n/cm^2 at 300 °C. Afterwards the samples were exposed to the electron beam in the Judith 1 installation at FZ. Jülich under conditions that simulate disruptions. The effect of combined neutron irradiation and electron beam exposure is studied with scanning (SEM) and transmission electron microscopy (TEM).

2. Experimental

The tungsten material studied here was obtained from a highly deformed and stress relieved tungsten bar provided by Plansee AG, Austria. Three discs of 10 mm diameter and 5 mm thick were produced. The surface of the discs is mechanically polished before further exposures. The three discs are labelled ref W, low dose and high dose. Sample ref W was kept as a reference. It was neither neutron irradiated nor exposed to the electron beam. The other two samples were neutron irradiated in the BR2 reactor of SCK·CEN at a temperature of 300 °C in a helium atmosphere. The low dose sample was irradiated to a neutron fluence (E > 1 MeV) of 1.47×10^{20} n/cm^2. The high dose sample was irradiated to a neutron fluence (E > 1 MeV) of 4.74×10^{20} n/cm^2. The dose levels with which these fluences correspond, depend on using the EFF 1.1 or the TENDL-2011 nuclear data library [5]. The doses are 0.07 dpa, respectively 0.17 dpa for the low dose sample and 0.24 dpa, respectively 0.53 dpa for the high dose sample. The amounts of transmutation products were calculated to be 0.56 mass% Re and 0.01 mass% Os in the low dose sample and 1.59 mass% Re and 0.04 mass% Os in the high dose sample.

After neutron irradiation, both samples were exposed to the electron beam of the JUDITH 1 facility at FZ Jülich ($P_{max} = 60$ kW, $U_{acc} = 120$ keV, beam width at half maximum = 1 mm). They were exposed to one pulse of 5 ms on an area of 4×4 mm^2 and the absorbed power density was 0.33 GW/m^2. To obtain a homogeneous loading pattern, a scanning frequency in the two orthogonal directions of $f_x = 40$ kHz and $f_y = 31$ kHz is applied. The base load temperature is at room temperature. These loading conditions are representative of a plasma disruption at the first wall [22]. It was measured that the surface temperature increased to 1520 K at the end of the pulse, after which the temperature drops to below 1100 K in 3 ms.

For this study, five samples were produced from the three discs. From the reference material, only a bulk sample was prepared. From the irradiated samples, one sample was prepared from the bulk and one sample was prepared close to the surface. The difficulty with the latter sample is that it needs to be prepared as close to the surface as possible. The sample preparation is schematically represented in Fig. 1. First a 1.5 mm thick disc is cut from the sample. Two laths are cut from this disc. A first lath (no. 1 in the figure) has a width of about 2 mm and contains the exposed area. The bottom part of this lath is removed by mechanical polishing on SiC paper until the thickness is reduced to 0.1 mm. A platelet was taken from the exposed part of the surface. The second lath (no. 2 in the figure) has a width of about 1 mm. This sample was flipped (top indicates the top surface from the disc) and mechanically polished from both sides to reduce the thickness to 0.1 mm. One platelet was broken off, giving the cross-section orientation.

The final step of the sample preparation was electrochemical polishing using a Struers Tenupol-5 instrument. The sizes of all platelets are of the order of 1.5–2 mm. The platelets were glued on a 3 mm copper 1 mm aperture grid to be able to mount them in a TEM holder. The polishing conditions were the same for all samples. The electrolyte consisted of 1.5 wt% NaOH in water and the voltage that was applied was 20 V. All bulk samples, from the reference material as well as the irradiated samples, were double jet polished until perforation. The two surface exposed samples were double jet polished for 10 s, to remove the surface roughness, after which polishing was continued from the back side only until perforation. It was not determined exactly how much material was removed from the exposed side, but it's not more than a few micrometers.

All samples were investigated in a JEOL 3010 microscope operating at 300 kV. Conventional bright field, dark field and weak beam imaging was used. The local thickness was determined from convergent beam electron diffraction (CBED) patterns.

3. Results

3.1. Reference sample ref W

The grain structure obtained from the TEM investigation is shown in Fig. 2. These images confirm that the crystal grains have a sub-grain structure. The difference in orientation between part of the grains is small and they are separated by small-angle grain boundaries, but other grains have a completely different orientation and they are separated by large-angle grain boundaries. Tungsten materials with a sub-grain structure, studied previously [23,24] did not have large-angle sub-grain boundaries and also the difference in grain orientation at the low-angle grain boundaries is larger in this material. In the diffraction pattern corresponding to the grain boundary of Fig. 2b, it can be seen that the diffraction spots of both grains are rotated over 6.5°. This value is much higher than in previously studied double forged tungsten, where the misfit angle was only 3°. The average size of the sub-grains is 1.6 μm.

Apart from the grain boundaries, very few defects are present. There are no defects visible in the interior of the grain and only near the grain boundaries some line dislocations can be observed. The dislocation line density near the grain boundaries is of the order of 1×10^{13}/m^2, but the total amount is too low for a more precise measurement. From the verification of the extinction conditions it can be concluded that most of the dislocations are a/2⟨111⟩ type. In general the Burgers vector is also parallel to the dislocation line, which means that they are screw dislocations. A minority of the line dislocations have an a⟨100⟩ Burgers vector. These dislocations are usually formed as a result of the interaction between two a/2⟨111⟩ type dislocations.

The observed defect structure is quite typical for a deformed and stress relieved tungsten material, which was not subjected to a recrystallization treatment. The stress relief annealing removes most of the defects in the grains but it doesn't remove the sub-grains as in higher temperature recrystallization treatments at 1500 K or above. The larger orientation difference between the sub-grains is probably the result of the high deformation in the tungsten rod.