Bend Stress Relaxation of Pure Tungsten

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Tungsten is one of the candidate materials being considered for fabricating the plasma facing components (PFCs) of fusion reactors. When used in fusion reactors, PFCs are exposed to high heat loads from the fusion plasma, resulting in the PFCs being subjected to induced thermal stresses. This study focused on the creep deformation of pure tungsten under such a deformation-controlled thermal stress. Bend stress relaxation (BSR) tests were performed on specimens of as-received pure tungsten at temperatures of 600 - 1000°C for 0.1 - 1 h in vacuum. The BSR ratio decreased with an increase in the temperature, decreasing sharply in a short period of 0.1 h and then decreasing gradually. Most of the stress was relieved during the BSR test performed at 1000°C for 1 h. The activation energy of stress relaxation, calculated using the cross-cut method, was similar to that of the Peierls mechanism. This suggested that the mobility of the dislocations controls the stress relaxation mechanism. Most of the stress was relieved quickly because the activation energy of stress relaxation was relatively small.

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1. Introduction

Tungsten is one of the candidate materials being considered for making the plasma facing components (PFCs) of fusion reactors. This is because of its high melting temperature, high thermal conductivity and high resistance to physical sputtering. When used in fusion reactors, PFCs are exposed to high heat loads from the fusion plasma. It is expected that the divertor of the International Thermonuclear Experimental Reactor (ITER) will be exposed to a maximum heat flux of 10 MW/m² and its maximum temperature will reach 1000°C [1,2]. Therefore, PFCs are subjected to thermal stresses owing to the thermal gradient between their plasma-facing and the cooling-channel sides. This is expected to result in stress relaxation owing to thermally activated creep under the influence of the deformation-controlled thermal stresses. Therefore, the creep deformation behavior of tungsten at high temperature needs to be elucidated.

The objective of this study was to investigate the effects of temperature and time on the stress relaxation behavior of pure tungsten at the temperatures encountered by PFCs in fusion reactors. The bend stress relaxation (BSR) method, which has been used to evaluate the creep properties of ceramics [3–8], was employed to determine the stress relaxation behavior of tungsten under deformation-controlled thermal stresses.

2. Experimental

Pure tungsten in the form of a rolled sheet, fabricated by Plansee Ltd., was used for the experiment. The purity of the pure tungsten was more than 99.97%. Test specimens having thicknesses of 0.10, 0.15, and 0.20 mm were fabricated from the rolled sheet. Specimens with three different thicknesses were prepared to investigate the effect of the maximum initial strain. The dimensions of the specimens were 40 mm length $\times 1.2$ mm width $\times 0.10$, 0.15, and 0.20 mm thickness. Figure 1 shows a schematic illustration of the fixture used to hold the specimens. All the fixture parts, which were made of β -silicon carbide, were fabricated by Rohm and Haas using chemical vapor deposition (CVD). The specimen was fixed using two loading



Fig. 1 Schematic illustration of the fixture used to perform the BSR test.

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Fig. 2 Measurement of the bend radii of the constrained and relaxed specimens.

plates having a radius of curvature of 100 mm. The specimen deformed bend mode and constant strain was loaded. The maximum initial strains applied to the surfaces of the specimens having thicknesses of 0.10, 0.15, and 0.20 mm were 0.03%, 0.05%, and 0.07%, respectively. The strains applied were within the elastic limit at room temperature. When elastically deformed specimens are heated to a high temperature, the elastic strain changes to permanent strain, resulting in relaxation of the loading stress. This phenomenon is called stress relaxation. This type of deformation is time dependent and similar to creep deformation.

The degree of stress relaxation was evaluated using the BSR ratio (m), which defined by the following equation [3].

$$m = 1 - R_{\rm o}/R_{\rm a} = 1 - \Phi_{\rm a}/\Phi_{\rm o},\tag{1}$$

where Φ_0 and Φ_a are the initial and final bend angles and $R_{\rm o}$ and $R_{\rm a}$ are the initial and final curvatures, respectively, of the specimen being tested, as shown in Fig. 2. A specimen is fully relaxed when the BSR ratio is equal to 0 and non-relaxed when the BSR ratio is equal to 1. For example, a fully relaxed specimen has the same curvature as that of the loading plate, while the shape of a non-relaxed specimen reverts to that of a flat plate. In this study, the BSR ratio was determined by measuring the bend angles $(\Phi_0 \text{ and } \Phi_a)$ using an optical microscope. The bend angle measurements were accurate to less than 0.3° . This gives a potential scatter less than 0.02 in the BSR ratio. The BSR test was carried out in vacuum with the pressure being 10^{-3} Pa. Oxidation of the surfaces of the specimens was not observed. The test temperatures and test durations were 600, 800, 900, and 1000°C, and 0.1, 0.5, and 1 h, respectively.



Fig. 3 The values of the BSR ratios (m) of the pure tungsten specimens plotted against the maximum initial tensile strain.

In order to investigate the mechanism of stress relaxation, the microstructures of the specimens after the BSR test at 900°C for 1 h in vacuum were observed using a transmission electron microscope (TEM, JEOL JEM-2010). The microstructures of unheated specimens and specimens heat-treated at 900°C for 1 h in vacuum but not subjected to a bending strain were also observed.

3. Results and Discussion

The BSR ratio, *m*, can be determined using the ratio of the initial stress (σ_0) and the residual stress (σ_a), which is given by following equation [3]:

$$m = \sigma_{\rm a}/\sigma_{\rm o} = (\varepsilon_{\rm o} - \varepsilon_{\rm c})E/\varepsilon_{\rm o}E = 1 - \varepsilon_{\rm c}/\varepsilon_{\rm o}, \qquad (2)$$

where E is the elastic constant, ε_c is the creep strain, and ε_0 is the initial strain. The amount of creep strain experienced by a specimen is considered to be proportional to the amount of initial strain that it undergoes at the same temperature and over the same duration. Thus, the BSR ratio, which is described by Eq. (1) and using the relations $\varepsilon_{\rm c} = z/R_{\rm o}, \, \varepsilon_{\rm o} = z/R_{\rm a}$ (where z is the distance from the bend neutral axis in the specimen as shown Fig. 2), can be calculated as a value independent of the amount of initial strain [3]. Figure 3 shows the values of the BSR ratios of the specimens tested at 600, 800, 900, and 1000°C for 0.1, 0.5, and 1 h. These values are plotted against the maximum initial tensile strain values. For the same test duration and the same test temperature, the values of the BSR ratios of the specimens were nearly constant, regardless of the initial strain. Thus, the results of this study also confirmed that BSR ratio does not depend on the initial strain.

Figure 4 shows the temperature dependence of the BSR ratios of the specimens tested at 600, 800, 900, and 1000°C for 0.1, 0.5, and 1 h, respectively. The BSR ratios decreased with an increase in the test temperature. However, even when the different test temperatures and test durations were different, the slopes of the curves were al-



Fig. 4 The values of the BSR ratios (m) of the pure tungsten specimens plotted against the reciprocal test temperature.



Fig. 5 Time dependence of the BSR ratio (m) of pure tungsten.

most the same. During the BSR test that was performed at 1000°C for 1 h, the loaded stress was completely relieved.

Figure 5 shows the curve of the values of the BSR ratios against the test durations. The stress decreased exponentially with an increase in the test duration. The BSR ratios decreased sharply for test durations lower than 0.1 h and then decreased slowly. The slopes of the curves in Fig. 5 were almost the same.

In order to investigate the mechanism of stress relaxation, the activation energy of stress relaxation was evaluated using the cross-cut method [9, 10]. Since stress relaxation occurred during a thermally activated process, the activation energy, Q, could be obtained from $\Delta(1/T)$ (where T [K] is the test temperature), the difference in the value of the reciprocal of temperature for a constant BSR ratio in Fig. 4. The value of Q for a given value of m can be determined using the equation given below.

$$Q = R \ln(t_2/t_1) / (1/T_1 - 1/T_2), \tag{3}$$

where R is the gas constant (8.314 J/(mol.·K)), t_1 and t_2 are the test durations and T_1 and T_2 are the test temperatures for t_1 and t_2 , respectively. When t_1 was 0.1 h and t_2 was 1 h, the activation energies at m = 0.2, 0.3, and 0.4were calculated to be 152, 154, and 164 kJ/mol, respectively. The activation energy of the Peierls mechanism is the periodic potential energy that a dislocation receives because of the periodic atomic arrangement, when it moves on a surface [11]. Therefore, the activation energy of the Peierls mechanism is considered to be the energy required by dislocations to move and is 150 - 200 kJ/mol in the case of tungsten [12, 13]. The activation energy of stress relaxation obtained from Fig. 4 was similar to that of the Peierls mechanism. On the basis of this result, it can be assumed that the stress relaxation mechanism might be controlled by the mobility of dislocations.

Figure 6 shows micrographs of the dislocations in the matrices and grain boundaries of the various specimens, obtained using transmission electron microscopy (TEM) under conditions where g = [200]. The micrographs are for (a) an as-received (unheated) specimen, (b) a specimen after heat treatment at 900°C for 1 h while not being subjected to a bend stress, and (c) a specimen after the BSR test at 900°C for 1 h. The difference in the dislocation densities of the matrices of the as-received specimen and the specimen subjected to the BSR test was not obtained. On the other hand, in the as-received specimen, dislocations that had accumulated near the grain boundaries were observed, while no dislocations were observed in the specimen subjected to the BSR test. This was because the dislocations near the grain boundaries were absorbed by the grain boundaries during the BSR test. That is to say, the recovery of the dislocations occurred. No differences were observed in the structures of the dislocations seen in the matrices and those near the grain boundaries. This was true for the specimen subjected to the BSR test and the specimen that was heat-treated while not being subjected to stress. This was because, in this study, the amount of maximum initial tensile strain experienced by the specimens during the BSR test was small and only 0.07%. However, it is believed that creep in metals at low temperatures (temperatures less than half their melting points) is not dominated by diffusion [14] but by cross slips, the crossing of forest dislocations, and the friction caused in the lattice by the Peierls force [15]. These observations suggested that, under the treatment conditions investigated in this study, the mobility of the dislocations controlled the stress relaxation behavior of pure tungsten.



Fig. 6 TEM micrographs of (a) an as-received specimen, (b) a specimen after heat treatment at 900°C for 1 h, and (c) a specimen after the BSR test at 900°C for 1 h.

The degree of stress relaxation might be highly dependent on the test temperature since the mobility of the dislocations, which control stress relaxation, increased with the test temperature. Most of the stress was relieved with a short period because the activation energy for stress relaxation was small. The degree of stress relaxation during the BSR test 0.1 h in duration was high since the dislocations, which were able to move easily owing to only a few obstacles being present, moved during the early stages of the test. Then, the degree of stress relaxation decreased relatively slowly because the dislocations were consumed. In addition, the extent of the movement of the dislocations decreased since the stress reduced with an increase in the test duration. In other words, the creep rate during stress relaxation decreased with time. Therefore, the stress was relieved slowly.

4. Conclusion

The BSR test was performed on as-received specimens of pure tungsten at temperatures of 600, 800, 900, and 1000°C for 0.1, 0.5, and 1 h in vacuum to investigate the effects of temperature and time on the stress relaxation behavior of pure tungsten under conditions encountered by PFCs in fusion reactors. The following results were obtained:

- The BSR ratio decreased with an increase in temperature. Initially, the BSR ratio decreased precipitously in a short period (≤ 0.1 h), and then decreased slowly. Most of the stress was relieved during the BSR test performed at 1000°C for 1 h.
- (2) The activation energy of stress relaxation, determined by the cross-cut method, was similar to that of the Peierls mechanism. This finding suggested that stress relaxation was controlled by the mobility of the dislocations. In addition, most of the stress was relieved within a short period because the activation energy of stress relaxation was relatively small.

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