Mechanical properties of tungsten: Recent research on modified tungsten materials in Japan

Shuhei Nogami , Akira Hasegawa , Makoto Fukuda , Michael Rieth , Jens Reiser , Gerald Pintsuk

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NMechanical properties of tungsten: Recent research on modified tungsten materials in Japan

Shuhei Nogami[a,*] shuhei.nogami.d3@tohoku.ac.jp, Akira Hasegawa[b] akira.hasegawa.d2@tohoku.ac.jp, Makoto Fukuda[c] fukuda.makoto@qst.go.jp, Michael Rieth[d] michael.rieth@kit.edu, Jens Reiser[e] jens.reiser@kit.edu, Gerald Pintsuk[f] g.pintsuk@fz-juelich.de

[a]Department of Quantum Science and Energy Engineering, Graduate School of Engineering, Tohoku University, 6-6-01-2, Aramaki-aza-Aoba, Aoba-ku, Sendai 980-8579, Japan

[b]Department of Quantum Science and Energy Engineering, Graduate School of Engineering, Tohoku University, 6-6-01-2, Aramaki-aza-Aoba, Aoba-ku, Sendai 980-8579, Japan

[c]Department of Quantum Science and Energy Engineering, Graduate School of Engineering, Tohoku University, 6-6-01-2, Aramaki-aza-Aoba, Aoba-ku, Sendai 980-8579, Japan (Present affiliation: National Institutes for Quantum and Radiological Science and Technology, 801-1, Mukouyama, Naka, Ibaraki 311-0193, Japan)

[d]Institute for Applied Materials, Karlsruhe Institute of Technology, Hermann-von-Helmholtz-Platz 1, 76344 Eggenstein-Leopoldshafen, Germany

[e]Institute for Applied Materials, Karlsruhe Institute of Technology, Hermann-von-Helmholtz-Platz 1, 76344 Eggenstein-Leopoldshafen, Germany

[f]Forschungszentrum Jülich GmbH, Institute for Energy and Climate Research, 52425 Jülich, Germany

*Corresponding Author.

Abstract

There remain some drawbacks of mechanical properties of W materials as a plasma facing material (PFM) for fusion reactor divertors, which are low temperature brittleness, high ductile-to-brittle transition temperature (DBTT), and recrystallization-induced embrittlement. To solve these issues, development of W materials with improved thermo-mechanical properties, neutron irradiation tolerance, and possibility of mass-production with microstructural uniformity has been advanced for the last decade under the collaboration R&D by universities in Japan. In this paper, the effects of grain refining, K-doping, dispersion strengthening by La_2O_3 particles, and alloying by Re are discussed from the viewpoints of both short- and long-term material properties and phenomena, including effects of neutron irradiation and high heat loads, which should be considered under the actual fusion reactor environments. Through this R&D, K-doping and Re-addition showed several positive effects. Among the materials developed in this R&D, K-doped W-3%Re hot-rolled plate could be a better solution for PFM, which demonstrated superior properties from several perspectives. However, materials alloyed by Re have an intrinsic concern of higher irradiation hardening caused by neutron irradiation up to higher doses. Therefore, it is pointed out that investigations of thermo-mechanical properties under higher dose neutron irradiation are significantly required to realize long-term structural reliability and lifetime of fusion reactors.

Keywords:

Tungsten, Mechanical property, Powder metallurgy, Dispersion strengthening, Alloying

1. Introduction

The estimated steady state heat flux in plasma facing materials (PFMs) of future fusion reactor divertors will be around 5-10 MW/m^2 [1, 2]. However, due to possible off-normal events, transient heat loads up to about 20 MW/m^2 or even higher have to be considered for safety reasons [2]. In the case of edge-localized mode (ELMs) discharges, the steady-state heat load will be overlaid by short pulses in the GW/m^2 range [3]. Depending on the specific operating scenario, there are different possible degradation processes, which will limit the lifetime of plasma-facing divertor components. For example, degradation of physical and mechanical properties of PFMs could occur due to surface modification, cracking, deformation, ageing, recrystallization, and melting [4–6]. In addition to heat load, PFMs will be damaged by neutron irradiation, which could include displacement damage (hardening, void swelling, radiation induced segregation or dissolution, etc.) as well as transmutation damage (phase formation due to transmutation products etc.) [7]. Most of these irradiation effects lead to embrittlement and other degradations of PFM properties. Therefore, the operating limits of divertor components have to be evaluated by considering heat load scenarios and neutron irradiation, simultaneously.

Pure tungsten (W) is a primary PFM candidate for high heat flux components in the divertor region of a fusion reactor because of its high melting point, thermal conductivity, sputtering resistance, and potentially low tritium retention [8]. However, there remain some drawbacks related to the thermo-mechanical properties, e.g., low temperature brittleness, high ductile-to-brittle transition temperature (DBTT) [9–17], recrystallization embrittlement [18–21], and neutron irradiation embrittlement [22–25]. The upper and lower limits of operation temperature of divertor components using W material would be determined by the recrystallization temperature and DBTT, respectively (see Fig. 1) [26]. However, the neutron irradiation could increase the DBTT [25] and has a possibility of changing the recrystallization temperature. Therefore, the operation temperature range determined by the recrystallization temperature and DBTT could be shrunk by the neutron irradiation.

To solve these issues, development of irradiation-tolerant W materials with improved thermo-mechanical properties has been advanced for the last decade under collaboration R&D by universities in Japan. Several modification techniques, e.g., grain refining [27], work hardening [28], alloying [29, 30], and dispersion strengthening [30, 31], have been considered with the aim to improve ductility and toughness even under neutron irradiation. There are several special production methods to obtain materials with excellent properties, e.g., ultra-fine grained (UFG) W–TiC compacts fabricated by powder metallurgical methods utilizing mechanical alloying (MA) [32], however, some methods were limited to a laboratory-scale material production so far. Therefore, a powder metallurgy was selected in this R&D to obtain W materials for mass-production with microstructural uniformity.

This paper summarizes current understanding and issues related to the thermo-mechanical properties of W materials developed in this R&D, which were fabricated by powder metallurgy and rolling/swaging. The results previously reported and the additional data newly obtained are shown in this paper. The effects of grain refining, dispersion strengthening by potassium bubbles (K-doping) [31, 33–35], dispersion strengthening by lanthanum oxide (La_2O_3) particles [36, 37], and solid solution alloying by rhenium (Re) [38–40] are discussed from the viewpoints of high-temperature microstructural stability (recovery, recrystallization, and grain growth), Charpy impact properties, and tensile properties (see sections 3, 4, and 5).

K-doping is a common dispersion-strengthening method for W materials and has been historically used for W filaments etc. [31, 33–35]. K-doped W contains nano-bubbles including K atoms (on the order of ppm), which are mainly dispersed at the grain boundaries [31]. K-bubbles are produced during sintering by the volatilization of K, which is added to the raw material powder. Because K-bubbles can hinder the motion of grain boundaries and dislocations, they lead to strengthening at a high temperature and suppression of recrystallization [33, 35]. In addition, K-doping can produce finer grains compared to pure W because K-bubbles inhibit grain boundary migration [35]. This grain refining also leads to strengthening and toughening [41]. The similar positive effects are expected by applying the dispersion of La_2O_3 particles [36, 37].

Alloying by Re is also a common method for solid solution alloying of W materials [38–40]. It is well known that substitutional solid solution elements in body-center-cubic (bcc) metals like Re in W could cause solid solution strengthening at high temperature and solid solution softening at low temperature. Solid solute Re in W materials could improve not only mechanical properties, but also resistance to recrystallization and neutron irradiation [42, 43]. Changes in the thermo-mechanical properties, resistance to recrystallization, and resistance to neutron irradiation by Re addition are dependent on the additive amount of Re [38–40, 43–45].

A long-term operation is expected in future fusion reactors like DEMO. Thus, an assessment of long-term structural reliability and lifetime is essential. As for the PFMs, time-dependent phenomena and properties, e.g., cyclic fatigue, creep, creep-fatigue interaction, ratcheting, accumulation of plastic-strain, long-term microstructural stability, and neutron irradiation effects, should be assessed. Therefore, evaluation results of fatigue life, long-term microstructural stability, and effects of neutron irradiation of pure and modified W materials developed in this R&D are also described in this paper (see section 6).

Furthermore, PFMs of fusion reactor divertors will suffer complicated heat loads, including steady state, transient, and ELM heat loads, as shown in Fig. 1. Under these heat load environments, integrated thermo-mechanical properties are important because various types of loadings would be simultaneously applied. Material related structural and lifetime limitations cannot be determined by considering the individual material properties but by assessing the synergistic load effects. Therefore, possibility of improving structural strength and lifetime of fusion reactor divertors by using the modified materials as PFMs instead of ordinary pure W is also discussed in this paper based on the results of heat load tests and structural analysis (see section 7).

2. Materials

In this R&D, K-doping, dispersion of La_2O_3 particles, and alloying by Re were mainly applied as modification methods of W material. To clarify the effects of these modifications, major conditions of fundamental production routes were the same for all materials, which included cold isostatic pressing, sintering, rolling or swaging, and finally a heat treatment for stress relief. To investigate the effect of reduction ratio (deformation ratio) in rolling and swaging, a few levels of deformation ratio were applied.

Plates made of pure W, K-doped W, W-3%Re, K-doped W-3%Re, and W-3%Re-1%La_2O_3 and rods made of pure W and K-doped W were fabricated in the present study. The concentration of K in K-doped W and K-doped W-3%Re was approximately 30 ppm. K-bubbles and La_2O_3 particles (see Figs. 2 (a) and (b)) are considered to be mainly dispersed at grain boundaries, hinder the motion of grain boundaries and dislocations, leading to finer grains, higher strength at high temperatures and suppression of recrystallization [33, 35]. The additive amount of Re (3%) in the present study was determined according to the knowledge of the changes in mechanical properties, thermal conductivity, resistance to recrystallization, and resistance to neutron irradiation by Re addition [46].

The plate materials were fabricated by powder metallurgy and hot rolling followed by a final heat treatment at 900°C for 20 min for stress relief. The deformation ratio for hot rolling was in two levels; higher level of deformation ratio (hereafter, indicated by “H”) and lower level of deformation ratio (hereafter, indicated by “L”). The thickness obtained directly after rolling was 7 mm for all materials. As shown in Figs. 3 (a) and (b) [47–50], flattened grain structures elongated along the rolling direction like a pancake were observed, which were characteristic for W plates fabricated by powder metallurgy and rolling [51]. As shown in Fig. 3 (c), a lot of sub-grains were observed inside of the grains defined by high-angle grain boundaries.

The rod materials were fabricated by powder metallurgy and swaging followed by a final heat treatment at 900°C for 20 min for stress relief. The deformation ratio for swaging was in three levels; higher level of deformation ratio (hereafter, indicated by “H”), intermediate level of deformation ratio (hereafter, indicated by “I”), and lower level of deformation ratio (hereafter, indicated by “L”). The diameters obtained directly after swaging were 6, 10, and 20 mm for “H”, “I”, and “L”, respectively. As shown in Figs. 4 (a~d), grains grew to needle-like shape elongated along axial direction of the rods with increase in the deformation ratio.

To investigate the chemical composition of materials used in the present study, an inductively coupled plasma atomic emission spectroscopy (ICP-AES) and an infrared absorption spectrometry (IR) were used for the analysis of metal and gas elements, respectively. The concentration of carbon (C), oxygen (O), and nitrogen (N) as interstitial impurities in all materials, except the W-3%Re-1%La_2O_3 plate, were less than 10 ppm. Because such interstitial impurities are known to influence recrystallization, grain growth, and the other related material properties due to their segregation at grain boundaries, lowering those concentrations was considered in the fabrication. From the viewpoint of concentration of impurity elements, it is required in ITER diverter that the concentration of C, O, and N in the pure W should be less than 0.01 % (100 ppm). Thus, the materials in the present study, except the W-3%Re-1%La_2O_3 plate, were satisfied with the requirements. The concentration of C, O, and N in the W-3%Re-1%La_2O_3 plate were less than 10 ppm, approximately 1780 ppm (includes O in the La_2O_3), and less than 10 ppm, respectively. However, since the concentration of O in this material is mostly derived from La_2O_3, the concentration level of C, O, and N in the W-3%Re-1% La_2O_3 plate could be acceptable against the above requirements.

As shown in Table 1 [47–50, 52], the grain size of materials with the same major chemical composition decreased with increase in deformation ratio. In addition, as for the materials fabricated under the same deformation ratio, grain size decreased by K-doping, dispersion of La_2O_3 particles, and Re-addition, which could be attributed to the hindering motion of grain boundaries and dislocations by K-bubbles, La_2O_3 particles, and solute Re during fabrication [27, 42]. These tendencies were applicable for both rolled plates and swaged rods.

As shown in Fig. 5 [53, 54], thermal conductivity decreased with increase in amount of Re, especially at low temperatures. Although K-doping showed almost no effect on thermal conductivity of rolled plate, K-doped W (L) rod showed relatively lower thermal conductivity compared to pure W (H) plate. This could be attributed to the insufficient sintering and swaging conditions, which remained pores and elongated K-bubbles (see Fig. 6 (a). Fig. 6 (b) might be a better case.).

3. High-temperature microstructural stability

To evaluate the high-temperature microstructural stability including recovery, recrystallization, and grain growth stages, Vickers hardness and grain size were measured after isochronal annealing at 1100–2300°C for 1 h. As shown in Figs. 7 (a), (c), and (d) [50], the general stages of annealing temperature dependences of hardness was as follows: the first, a microstructural recovery stage, with a gradual decrease of hardness by around 10–20 HV from the respective as-received values with increase in the annealing temperature, the second, the primary recrystallization stage, with a rapid decrease by around 70–100 HV from the values after the recovery stage, and the third, further gradual decrease by around 10 HV up to 2300°C. From the viewpoint of these annealing temperature dependences of Vickers hardness, the recrystallization temperature by the isochronal annealing for 1 h of pure W (H) plate was 1250°C; K-doped W (H) plate was 1350°C; W-3%Re (H) plate was 1500°C; K-doped W-3%Re (H) plate was 1450°C; W-3%Re (L) plate was 1400°C; K-doped W-3%Re (L) plate was 1550°C; W-3%Re-1%La_2O_3 (L) plate was 1550°C; pure W (H) rod was 1400°C; K-doped W (H) rod was 1600°C; pure W (L) rod was 1700°C; and K-doped W (L) rod was 1700°C (see table 2). The Kernel average misorientation (KAM) could intrinsically indicate the average of differences in grain orientation. Thus, worked materials with a high density of dislocations and recrystallized materials with a very low density of dislocations could show high and low KAM values in general, respectively. Based on the KAM images shown in Fig. 7 (b), the recrystallization temperature of pure W (H) plate, K-doped W (H) plate, and K-doped W-3%Re (H) plate were 1200–1300°C, 1300–1400°C, and 1400–1500°C (blue color means low KAM), respectively, which agreed with the recrystallization temperature determined by hardness measurement, as described above. It is well known that an increase in deformation ratio could produce relatively rapid microstructural recovery and low resistance to recrystallization in general [55, 56]. In the present study, the materials with higher deformation ratio showed slightly lower recrystallization temperature, compared to those with lower deformation ratio with the same major chemical compositions.

These results demonstrated a suppression of recrystallization by K-doping, dispersion of La_2O_3 particles, and Re-addition, which could be attributed to hindering the motion of grain boundaries and dislocations by the K-bubbles, La_2O_3 particles, and solute Re [27, 42]. In addition, the Re-addition decreased hardness in the as-received condition because of solid solution softening, whereas the K-doping and dispersion of La_2O_3 particles increased that. Furthermore, K-doped W-3%Re (L) and W-3%Re-1%La_2O_3 (L) plates indicated synergistic positive effects of Re-addition and the second phase dispersions (K-doping and dispersion of La_2O_3 particles) on suppression of recrystallization. In contrast, only a swaged K-doped W rods (L) showed no positive effect of K-doping on suppression of recrystallization. This could be attributed to the insufficient sintering and swaging conditions to obtain non-desired number density and size of K-bubbles (see Fig. 6 (a)).

As for the plate materials, grain growth during microstructural recovery and recrystallization was not significant except pure W (H) plate, as shown in Fig. 8 [50]. A significant grain growth occurred at 1200–1300°C and above 2000°C in pure W (H) plate, which could be attributed to the primary and the secondary recrystallizations, respectively. The data scatter of grain size was relatively large at 1900–2000°C. This could be attributed to that the significant grain growth could start at this temperature range and that each data point in Fig. 8 was obtained from the different samples that only experienced the one temperature condition. The other materials showed small grain growth during the primary recrystallization. W-3%Re (H) and K-doped W-3%Re (H) plates showed slight grain growth during the secondary recrystallization. In contrast, no clear grain growth occurred in K-doped W (H) and W-3%Re-1%La_2O_3 (L) plates. Based on these results, K-doping, dispersion of La_2O_3 particles, and Re-addition had an ability to suppress grain growth. Especially for grain growth caused by the secondary recrystallization at relatively high temperature, K-doping and dispersion of La_2O_3 particles were more effective than Re-addition. Most of the favorable effects of the second phase dispersions are known to

occur at high temperatures, e.g., above half of the melting temperature of matrix material. For example, high temperature creep deformation could be suppressed by the suppression of grain boundary sliding due to the second phase dispersions. Therefore, it was considered that the K-doping and dispersion of La_2O_3 particles could play roles effectively against phenomena occurring at relatively high temperatures, compared to the other modification methods such as alloying. As one example, it was considered that the suppression of grain growth was observed in the present study.

4. Charpy impact properties

It is well known that DBTT can be evaluated by several test methods, e.g., tensile test, bending test, Charpy impact test, and fracture toughness test. DBTT is strongly dependent on test method, strain rate, and specimen shape. In the present study, Charpy impact and tensile tests were performed to evaluate the DBTT.

Charpy impact tests were performed based on the EU standard using a KLST Charpy V-notched specimen along L-S (for plates) and L-R (for rods) directions at temperatures ranging from 200 to 1000°C in vacuum (the first letter (L): the direction perpendicular to the expected crack plane, the second letter (S and R): the expected direction of crack growth) [57].

4.1. Properties in the as-received condition

As shown in Fig. 9 [47, 51, 58–62] and Fig. 10, DBTT and upper shelf energy (USE) were varied widely with materials, even if their major chemical compositions were the same, which experienced different fabrication methods and histories (e.g., deformation ratio). Most materials showed a brittle fracture and a mixture of brittle and delamination fractures below DBTT and a delamination fracture above DBTT. In contrast, K-doped W rods showed a ductile deformation with small or no cracks above 800°C. As shown in Fig. 12 [47–51, 58–62], a Hall–Petch-type relation was obtained for DBTT vs. grain size (d_S) and USE vs. grain size (d_S). Thus, those varied DBTT and USE, which could not be determined only by the major chemical composition, might be attributed to the individual particular grain structure dependent on the fabrication methods and histories.

As shown in Fig. 11 [47–50], DBTT of pure W (H) plate was 550°C; K-doped W (H) plate was 350°C; W-3%Re (H) plate was 450°C; W-3%Re (L) plate was 550°C; K-doped W-3%Re (H) plate was 250°C; and W-3%Re-1%La_2O_3 (L) plate was 550°C (see table 3). As for the high-deformed materials (“H”), approximately 200 and 100°C reduction in DBTT and approximately 40 and 30% increase in USE were caused by the K-doping and Re-addition, respectively. In contrast, W-3%Re (L) and W-3%Re-1%La_2O_3 (L) plates showed very low absorbed energy, compared to the high-deformed materials. No significant positive effects by dispersion of La_2O_3 particles were observed in the low-deformed materials. Appearances of tested specimens shown in Fig. 11 indicated that delamination of low-deformed materials quickly propagated accompanied by almost no plastic deformation of base-metal, whereas high-deformed materials showed delamination accompanied by enough bending (plastic deformation). These results demonstrated Charpy impact properties could be improved by K-doping and Re-addition, when enough deformation was applied in rolling and swaging. Moreover, synergistic effects of K-doping and Re-addition were clearly observed. In contrast, effect of dispersion of La_2O_3 particles has to be clarified in future work by applying to the high-deformed materials.

As well as the pure W, Hall–Petch-type relations between DBTT, USE, and grain size (d_S) could also fit the DBTT and USE of K-doped W (H), as shown in Fig. 12 [47–51, 58–62]. The effect of K-doping could be roughly distinguished as the effects of grain refining and dispersion of K-bubbles. On the other hand, the Hall–Petch-type relations included only the factor of grains. Therefore, the dispersion of K-bubbles could not directly influence the Charpy impact properties below 1000°C, although the improvements in the impact properties due to grain refining caused by K-doping were significant. Because the effect of K-bubbles was originally expected, especially at higher temperatures such as the temperature ranges showing creep, the impact properties at much higher temperatures should be investigated to clarify the effect of K-doping in further detail. In the case of W-3%Re (H) and K-doped W-3%Re (H) plates, the experimentally determined DBTT and USE were slightly lower and higher than those expected by the Hall–Petch-type relations for the pure W materials and K-doped W (H) plates, as shown in Fig. 12 [47–51, 58–62]. The effect of Re-addition could be roughly consist of the effects of grain refining caused by the inhibition of the grain boundary migration by the solute Re and the effects of solid solution strengthening and softening caused by the solute Re. Therefore, the improvement of Charpy impact properties of W due to the Re-addition could be attributed to both grain refining and solid solution strengthening and softening. Thus, it is possible that the DBTT and USE could be roughly determined by grain size and effect of Re-addition, below the

temperature of 1000°C. In contrast, W-3%Re (L) and W-3%Re-1%La_2O_3 (L) plates did not obey the relations. This could be attributed to the insufficient deformation ratio in rolling to obtain desired grain matrixes (number density and size of sub-grains and dislocations, etc.) and grain boundaries (strength and feature, etc.).

As for the high-deformed rolled plates below DBTT, pure W (H) plate showed a cleavage fracture, whereas K-doped W (H), W-3%Re (H), and K-doped W-3%Re (H) plates showed an intergranular fracture at sub-grain boundaries, as shown in Figs. 13 (a) and (b) [48, 50]. In contrast, the intergranular fracture at sub-grain boundaries were observed in all four materials above DBTT. According to the report by Curry and Knott [63], cleavage fracture stress could increase with grain refining. Therefore, one of the reasons for suppression of cleavage fracture in K-doped W, W-3%Re, and K-doped W-3%Re could be grain refining compared to pure W.

According to these Charpy impact test results of as-received materials along L-S direction, the DBTT and USE were improved by K-doping and Re-addition if the deformation ratio in rolling was sufficient. In contrast, as for the low-deformed materials, no significant positive effects by dispersion of La_2O_3 particles were observed. However, there is an anisotropy of grain structure in the rolled plates and swaged rods, as mentioned in section 2, which could also result in the anisotropy of Charpy impact properties. For examples, Rieth *et al*. [61, 62] and Reiser *et al*. [58] reported the anisotropy of Charpy impact properties of rolled plates and round-blanks of pure W and W alloys. In their experiments, some materials showed significantly low absorbed energies along directions other than the L-S direction. Therefore, further evaluations of the anisotropy of Charpy impact properties are planned as a future work to fully understand the effectiveness of modification by K-doping, dispersion of La_2O_3 particles, and Re-addition.

4.2. Properties after recrystallization

It is known that mechanical properties of W materials could be changed by microstructural recovery, recrystallization, and grain growth. As mentioned in section 3, high-temperature microstructural stability could be changed by K-doping, dispersion of La_2O_3 particles, and Re-addition. Thus, the effect of annealing on Charpy impact properties of pure W (H), K-doped W (H), K-doped W-3%Re (H), and W-3%Re-1%La_2O_3 (L) plates were investigated. For this investigation, isochronal annealing was carried out at 1100, 1400, and 2300°C for 1 h before Charpy impact tests. According to section 3, temperatures of 1100 and 2300°C are below and above recrystallization temperatures for all four materials, respectively. In contrast, temperature of 1400°C is slightly above recrystallization temperatures of pure W (H) and K-doped W (H) plates and slightly below those of K-doped W-3%Re (H) and W-3%Re-1%La_2O_3 (L) plates.

The annealing at 1100°C produced no changes of absorbed energy (see Fig. 14 (a)). In contrast, the annealing at 2300°C produced zero absorbed energy (see Fig. 14 (c)) and intergranular fractures (see Fig. 15). As for the test results after the annealing at 1400°C (see Fig. 14 (b)), pure W (H) and K-doped W (H) plates, which were recrystallized before testing, showed brittle fractures with zero absorbed energy. In contrast, K-doped W-3%Re (H) and W-3%Re-1%La_2O_3 (L) plates, which were not recrystallized before testing, showed no degradation of absorbed energies, except the testing at 300°C of K-doped W-3%Re (H), where absorbed energy was still above zero. These results indicated that the microstructural recovery with no recrystallization could not influence Charpy impact properties and that the recrystallization could produce brittle intergranular fracture with zero absorbed energy, even at 1000°C.

4.3. Tensile properties

To investigate the tensile properties (ultimate tensile strength (UTS), 0.2% proof stress ($\sigma_{0.2}$), uniform elongation (UE), total elongation (TE), and reduction in area (RA)), tensile tests along L, T, and S directions for rolled plates and L and R directions for swaged rods were carried out at temperatures ranging from room temperature to 1300°C in vacuum using two kinds of flat-plate specimens (SS-J and VS-T type specimens) [48, 64]. The VS-T type specimen was only used for the tensile tests of rolled plates along S direction. A strain rate of 1×10^{-3} s^{-1} was applied for most tests and that of 3×10^{-5}, 1×10^{-2}, and 1×10^{-1} s^{-1} was applied for the tests to investigate the strain rate effect. Because cross-head displacement was controlled in tensile tests, values of strain shown in this paper (cross-head displacement / gauge length) would be larger than the actual strain in gauge section. The RA was

calculated from SEM images of the fracture surfaces. The number of tests at each temperature was one or two. The UTS, $\sigma_{0.2}$, UE, TE, and RA were evaluated using the average value if the number of tests was two.

5. Properties in the as-received condition

For the strength of rolled plates (see Figs. 16 (a, b) [48–50]), positive effects of K-doping, dispersion of La_2O_3 particles, and Re-addition were observed, whereas the effect of difference in deformation ratio (H vs. L) was small. It is known that grain refining can improve the strength of most metals including W [27, 41], even if their major chemical compositions are the same. In addition, it is also known that the K-doping, dispersion of La_2O_3 particles, and Re-addition could cause strengthening of W, especially at high temperatures. Therefore, grain refining by those modifications and strengthening by K-bubbles, La_2O_3 particles, and solute Re could induce higher strength of K-doped W, W-3%Re, K-doped W-3%Re, and W-3%Re-1%La_2O_3 compared to pure W. Significant decreases in strength of pure W (H) and K-doped W (H) were observed at temperature ranging from 1100 to 1300°C. The recrystallized W materials are known to show lower strength and higher elongation compared to the worked and stress-relieved W materials [40, 65]. The dislocation density in recrystallized grains might be very low and the lack of dislocation could induce decrease in strength and increase in elongation. As described in section 3, recrystallization temperatures of pure W (H) and K-doped W (H) plates were 1250°C and 1350°C, respectively. In contrast, recrystallization temperatures of the other materials were above 1300°C. Therefore, relatively low strength of these two materials at 1300°C could be attributed to the recrystallized grains with a very low density of dislocations caused by annealing.

As for the ductility (elongation and reduction in area) of rolled plates (see Figs. 16 (c~h) [48–50]), positive effects of K-doping, dispersion of La_2O_3 particles, and Re-addition were limited. The materials alloyed by Re (W-3%Re, K-doped W-3%Re, and W-3%Re-1%La_2O_3) showed higher UE and lower RA compared to the non-alloyed materials. It was reported that the material showing higher strain hardening could produce lower RA, which is quantitatively described by the work hardening coefficient. In addition, the material with higher work hardening coefficient could produce higher UE [66]. Thus, the lower RA of materials alloyed by Re compared to non-alloyed ones could be explained by the increase in work hardening coefficient accompanied by the increase in UE. Significant increases in TE and UE of pure W (H) plate were observed at temperature ranging from 1100 to 1300°C. Similar to the strength, this could be related to the differences in recrystallization behavior.

In the case of K-doped W swaged rods (see Figs. 17 (a, b) [52, 67]), relatively low strength and high total elongation compared to rolled plates were observed, especially for the K-doped W (L) rod. Although differences in UTS and TE between K-doped W (H and I) rods were small, K-doped W (L) rod showed significantly low UTS and high TE compared to these two materials, which was a similar trend to the DBTT by Charpy impact tests (see Fig. 10). Considering that the effects of difference in deformation ratio on tensile properties of the rolled plates (H vs. L) and swaged rods (H vs. I) were small, insufficient sintering and swaging conditions of the K-doped W (L) rod could be a reason of such low UTS and high TE.

$DBTT_{Tensile}$ was estimated based on the test temperature dependences of TE, where TE changed from a finite value to zero, as summarized in table 4. $DBTT_{Tensile}$ of pure W (H) plate was 150°C; K-doped W (H) plate was 50°C; W-3%Re (H) plate was 100°C; W-3%Re (L) plate was 100°C; K-doped W-3%Re (H) plate was below R.T.; K-doped W-3%Re (L) plate was 50°C; and W-3%Re-1%La_2O_3 (L) plate was 50°C. Approximately 50–100, 50, and 50°C reduction in $DBTT_{Tensile}$ was caused by K-doping, dispersion of La_2O_3 particles, and Re-addition, respectively. No significant effects of increase in deformation ratio were observed.

According to the tensile test results of as-received materials along L direction, strength (UTS and $\sigma_{0.2}$) and $DBTT_{Tensile}$ were improved by K-doping, dispersion of La_2O_3 particles, and Re-addition, whereas their effects on ductility (TE, UE, and RA) were limited and small. The effect of deformation ratio on tensile properties was less than its effect on the Charpy impact properties. However, there is an anisotropy of grain structure in the rolled plates and swaged rods, as mentioned in section 2, which could also result in anisotropy of tensile properties. As shown in Figs. 18 (a~f), Fukuda *et al*. reported the anisotropy of tensile properties of pure W (H), K-doped W (H), and K-doped W-3%Re (L) plates, which were the same materials of the present study [68]. Anisotropy of UTS was observed below approximately 500°C regardless of K-doping and Re-addition. All three materials showed the highest, intermediate, and lowest UTS along L, T, and S directions below 500°C, respectively. These differences increased with decrease in test temperature. As for the TE, anisotropy was also observed, especially at low temperatures. However, it was not clear in the K-doped W-3%Re (L) plate because of the scatter of TE values. This could be attributed to the insufficient deformation ratio in

rolling. For the case of swaged rods, Nogami *et al*. reported the anisotropy of tensile properties of pure W (L) and K-doped W (L) rods, as shown in Figs. 18 (g~j), which were also the same materials of the present study [67]. As well as the rolled plates, anisotropy of UTS and TE was observed, especially at low temperatures (below 500–700°C for UTS and below 700–1100°C for TE). The anisotropy of TE of low-deformed swaged rods was more significant than that of rolled plates. This could be attributed to the insufficient deformation ratio in swaging. Even for the low-deformed materials, K-doping made the low temperature ductility better, especially along radial direction. The anisotropy of mechanical properties should be considered not only in the material development phases but in the structural design phases of fusion reactors. One of the attempts of structural strength evaluation of divertor was reported by Fukuda *et al*. [69], where the anisotropy of strength was considered in the finite element analysis.

5.1. Properties after recrystallization

As well as the Charpy impact properties, the effect of annealing on tensile properties of pure W (H), K-doped W (H), K-doped W-3%Re (H), and K-doped W-3%Re (L) plates were investigated by tensile tests along T direction. For this investigation, isochronal annealing was carried out at 1500 and 2300°C for 1 h before tensile tests. According to section 3, the temperature of 2300°C is above recrystallization temperature for all four materials. In contrast, the temperature of 1500°C is slightly above recrystallization temperatures of pure W (H), K-doped W (H), and K-doped W-3%Re (H) plates and slightly below that of K-doped W-3%Re (L) plate.

As shown in Fig. 19, decrease in UTS and increase in TE occurred in all four materials by annealing at 1500 and 2300°C. There was not much difference in the results of the two annealing temperatures. Increases in $DBTT_{Tensile}$ were roughly 100−300°C in pure W (H) and K-doped W (H) plates. In contrast, the K-doped W-3%Re (H and L) plates showed no significant change of $DBTT_{Tensile}$. For the case of 1 h annealing at around recrystallization temperature (1400°C in the Charpy impact tests and 1500°C in the tensile tests), the effect of annealing on $DBTT_{Tensile}$ could be small compared to $DBTT_{Charpy}$. For the case of 1 h annealing at 2300°C, where the materials fully recrystallized, the effects were substantially different. The $DBTT_{Charpy}$ was severely affected by the full recrystallization, while the $DBTT_{Tensile}$ indicated only moderate variation. Thus, different sensitivity in the Charpy impact and tensile tests could be pointed out based on these evaluation results. This might be mainly caused by the difference in strain rate and the other factors, e.g., specimen shape and test method. The effects of strain rate on ductile-to-brittle transition behavior are discussed in the next section.

5.2. Effect of strain rate

As mention in section 1, divertor components are exposed not only to steady state heat loads but also to thermal shocks of extremely short duration, such as those produced during transient events. Therefore, divertor components are expected to experience mechanical loads at various temperatures and strain rates. Mechanical properties of bcc metals, including W, significantly depend on temperature and strain rate [70–72]. Thus, to evaluate the applicability of modified materials as fusion reactor materials, it is necessary to understand the test temperature and strain rate dependences of the mechanical properties. In addition, DBTT is one of the most important factors of W materials for divertor applications, however, it is strongly dependent on test method, strain rate, and specimen shape. In sections 4 and 5.1, Charpy impact and tensile tests showed significant differences in DBTT of pure and modified W materials, where one of the reasons could be a difference in strain rate. In this section, effects of strain rate on tensile properties of pure W (H), K-doped W (H), W-3%Re (H), and K-doped W-3%Re (H) plates and K-doped W (H and L) rods were discussed based on the results of tensile tests along L direction at strain rate ranging from 3×10^{-5} to 1×10^{-1} s^{-1}.

Strain rate dependences of $\sigma_{0.2}$ of pure W (H) plate in the as-received condition are shown in Fig. 20 [73]. As well as the pure W (H) plate, $\sigma_{0.2}$ of the other five materials increased with increase in strain rate and decrease in test temperature. The amount of change in $\sigma_{0.2}$ decreased with increase in test temperature at all strain rates. To evaluate these changes quantitatively, strain rate sensitivity (m) was evaluated, which is defined as the following equation [74]:

$$m = \partial \cdot \mathrm{Ln}\left(\sigma_{0.2}\right) / \partial \, \mathrm{Ln}(\dot{\varepsilon}) \qquad (1)$$

where $\dot{\varepsilon}$ is a strain rate. The value of m corresponds to a slope of each line in Fig. 20. As shown in Fig. 21 [73, 75], the strain rate sensitivity of all six materials was higher at lower temperatures and decreased with increase in test temperature. A similar trend of

pure W was found by Raffo *et al*. [38]. Strain rate dependences of TE at room temperature, 100, 200, and 300°C of pure W (H) plate in the as-received condition are shown in Fig. 22 [73]. At test temperatures of 100 and 300°C, small or no change of TE due to strain rate was observed. In contrast, a significant decrease in TE at strain rate of 10^{-3}–10^{-2} s^{-1} was observed at test temperature of 200°C. These results indicated the strain rate dependence of DBTT. In this case, $DBTT_{Tensile}$ of pure W (H) plate under tensile tests at strain rate of 10^{-5}–10^{-3} s^{-1} and 10^{-2}–10^{-1} s^{-1} were 100−200°C and 200−300°C, respectively. Similar tendencies were observed in the other modified materials [73, 75]. Because the difference in DBTT between Charpy impact and tensile tests was approximately 500°C in the maximum (W-3%Re-1%La_2O_3 (L) plate), not only strain rate but also the other factors, e.g., specimen shape and test method, have to be considered to fully understand the mechanism.

6. Mechanical properties for long-term operation

In sections 3, 4, and 5, material properties change and phenomena occurring during relatively short time are discussed (microstructural stability during relatively short time annealing, impact properties, and static strength and ductility). However, a long-term operation is expected in future fusion reactors like DEMO. Therefore, an assessment of long-term structural reliability and lifetime is essential for every component and material of these reactors. As for the PFMs of future fusion reactor divertors, time-dependent phenomena and properties, e.g., cyclic fatigue, creep, creep-fatigue interaction, ratcheting, accumulation of plastic-strain, long-term microstructural stability, displacement damage and transmutation by neutron irradiation, should be assessed to realize the long-term structural reliability and lifetime. However, lack of enough experimental data and knowledge is a significant issue for W materials as PFMs at present, especially for the recently developed modified materials. In this section, evaluation results of fatigue life, long-term microstructural stability, and effects of neutron irradiation of pure and modified W materials are described.

6.1. Fatigue life

Fatigue, e.g., thermal fatigue, low cycle fatigue, high cycle fatigue, and creep-fatigue interaction, produced by cyclic high heat flux loadings is one of the most important phenomena of PFMs of fusion reactor divertors. Analysis and prediction of structural strength and lifetime of divertors with consideration of fatigue have been conducted, e.g., reported by Li and You *et al*. [76]. For such analyses and predictions, the experimental data on fatigue life is essential, however, the number of experimental data has been very limited. Representative data have been reported by Schmunk *et al*. [77, 78] and Habainy *et al*. [79, 80].

As shown in Fig. 23, low cycle fatigue life of as-received and annealed pure and modified W materials (L direction) was evaluated in the present study by using a test apparatus and method developed under the IFMIF/EVEDA project [81–84], which was performed at room temperature and 500°C in vacuum at a strain rate of 0.1%/s. The temperature of 500°C was selected because DBTT of pure W was close to this temperature. A round-bar specimen with a test section diameter of 1.2 mm was used for the tests. Detailed test methods are reported in the previous papers [85, 86]. Almost no plastic strain could be applied in the case of fatigue tests at room temperature because plastic deformation capability of W materials is very low. As-received and recrystallized materials showed cleavage and intergranular fractures after room temperature tests, respectively (see #1 and #2 in Fig. 23). The effect of test temperature difference ranging from 500 to 1232°C seemed to be small in pure W materials. Modified W materials showed no significant improvement compared to pure W, except the possibility of life extension by K-doping at low strain range. Recrystallization produced slightly short fatigue life at higher temperature and significantly short fatigue life at room temperature, compared to the as-received materials. Although recrystallized materials also showed intergranular fracture even after the tests at 500°C, slip lines were observed on the fracture surface, which could indicate the plastic deformation capability even after the recrystallization (see #3 in Fig. 23). Further evaluations at widely-ranged test temperatures are planned as a future work to clarify the effectiveness of modification by K-doping, dispersion of La_2O_3 particles, and Re-addition.

6.2. Long-term microstructural stability

In section 3, the effects of modification by K-doping, dispersion of La_2O_3 particles, and Re-addition on high-temperature microstructural stability under relatively short time isochronal annealing were discussed. However, those microstructural changes are dominated not only by annealing temperature but also by annealing time. Pantleon *et al*. have shown behaviors of microstructural recovery and recrystallization of various pure W materials [87–93] during isochronal and isothermal annealing at temperature ranging

from 1100 to 1400°C for up to 3000 h. According to their results, deformation ratio was the other dominant factor for the high-temperature microstructural stability, as well as the ordinary metals [55, 56].

In the present study, effects of isothermal annealing on the high-temperature microstructural stability of modified W materials were investigated. Isothermal annealing experiments were performed at 1100°C for a maximum of 3000 h, which is below the 1 h recrystallization temperature of pure and modified W materials in the present study. As shown in Fig. 24 (a), recrystallization times of pure W (H), K-doped W (H), W-3%Re (H), and K-doped W-3%Re (H) plates were 10–100 h, 100–500 h, above 3000 h, and above 3000 h, respectively. The IPF images also indicated microstructure stability of K-doped W-3%Re (H) plate, even after the 3000 h annealing (see Fig. 24 (b)). These results demonstrated that the effectiveness of K-doping and Re-addition was clear and that of Re-addition was much higher than K-doping. Because an intrinsic positive effect of K-doping is expected at much higher temperatures, it is possible that the annealing at temperature above 1100°C might produce further positive effect of K-doping on microstructural stability. Thus, evaluations at widely-ranged test temperatures and longer time are planned as a future work to clarify the effectiveness of modification by K-doping, dispersion of La_2O_3 particles, and Re-addition.

6.3. Effects of neutron irradiation

In addition to heat loads, PFMs will be damaged by neutron irradiation, which could include displacement damage with solid and gas transmutations. Irradiation damages could induce microstructural change, thermo-mechanical and physical properties, which are important for PFMs of divertors. In this section, neutron irradiation responses of W materials are summarized, where data of modified W materials in the present study are included.

6.3.1. Irradiation hardening

Study on the irradiation-induced microstructure development, irradiation hardening, and effects of solid transmutation has been advanced compared to the other issues related to the neutron irradiation effects, which were investigated after neutron irradiation at temperatures up to around 800°C up to a few dpa using fission test reactors [7, 43, 94–112]. As shown in Fig. 25 (a) [43, 94, 95, 97–99, 103], irradiation hardening of pure W materials increased with displacement damage up to approximately 0.5 dpa, which were irradiated in Joyo (fast breeder reactor), JMTR (mixed-spectrum fission reactor), and HFIR (mixed-spectrum fission reactor). According to the microstructural analyses, irradiation hardening of pure W in this dose range could be mainly attributed to the void formation. Significantly higher irradiation hardening was produced above this irradiation dose in JMTR and HFIR with no thermal neutron shielding (HFIR (NS)), compared to Joyo and HFIR with thermal neutron shielding (HFIR (S)). Thermal neutrons could cause solid transmutations via (n, γ) neutron capture reactions, resulting in accumulation of Re, Os, and Ta, which could result in the formation of precipitates and clusters, inducing significant irradiation hardening [7, 43, 94–96, 99, 101, 103–109]. Because thermal neutrons can be negligible in Joyo and HFIR (S), such significant irradiation hardening in the presence of thermal neutrons could be caused by the formation of precipitates and clusters. In contrast, irradiation hardening saturated against the irradiation dose above approximately 0.5 dpa in Joyo and HFIR (S), which could be due to the void lattice formation. Void lattice formation has been considered to stabilize damaged microstructure by neutron irradiation. According to the irradiation study using accelerator-based ion-irradiation and nano-indentation techniques, the saturation of irradiation hardening was maintained up to 8 dpa (see Fig. 26 [54, 112]). The irradiation defects in pure W ion-irradiated up to 1 and 5 dpa at 800°C were dislocation loops and voids, which were not so different in number density and size. On the other hand, the irradiation defects in pure W irradiated up to 0.2 dpa were also dislocation loops and voids, but their number density and size were smaller than those of the irradiated up to 1 and 5 dpa. With increasing irradiation dose, the formation, recombination, and annihilation of the irradiated defects would simultaneously occur, and it could be considered that the amount of irradiation hardening was saturated due to their equilibrium.

In the case of W-Re binary alloys, especially alloys containing a few percent of Re (W-3%Re and W-5%Re), irradiation hardening below approximately 0.5 dpa was lower than that of pure W, as shown in Fig. 25 (b) [43, 94, 95, 97–99]. According to the microstructural analyses, suppression of irradiation hardening by Re-addition could be caused by the suppression of void formation due to solute Re. However, no suppression of irradiation hardening was observed above approximately 0.5 dpa because precipitations could start to form in the W-Re alloys. In contrast, W-Re binary alloys containing relatively high concentration of Re (W-10%Re and -26%Re) showed higher irradiation hardening compared to pure W, W-3%Re, and W-5%Re, which could be attributed to the formation of highly dense precipitations. In the case of W-Re alloys irradiated in Joyo, the amount of irradiation hardening by

irradiation up to approximately 1 dpa was lower than that by irradiation up to approximately 1.5 dpa. The kinds, number density, and size of irradiation defects were different between those two irradiation conditions [99]. Quantitative analysis of the contribution of each irradiation defect to the amount of irradiation hardening has not been carried out so far. In addition, irradiation defects, which are invisible by an TEM, may also contribute to the irradiation hardening [104]. Therefore, it is necessary to clarify the quantitative relationship between irradiation hardening and irradiation defects in future studies. Based on these irradiation studies of W-Re alloys, effect of Re-addition on irradiation hardening could be positive against low doses. However, if considering the production of high concentration of transmutant Re by long-term operation and utilizing W-Re binary alloys containing relatively high concentration of Re, relatively higher irradiation hardening has to be managed.

In the case of dispersion strengthened W materials, data of irradiation responses are limited. Related to the present study, K-doped W, K-doped W-3%Re, and W-1%La_2O_3 irradiated in Joyo and HFIR (S) up to around 0.5 dpa showed no clear effects of K-doping and dispersion of La_2O_3 particles on irradiation hardening and microstructure development, as shown in Fig. 25 (c) [100, 102, 110, 111]. This tendency was also observed up to 8 dpa by studies using ion-irradiation and nano-indentation techniques (see Fig. 26 [54, 112]).

6.3.2. Macroscopic mechanical properties

As for the macroscopic mechanical properties, e.g., DBTT, strength, ductility, fatigue life, and creep deformation, these data are further limited. According to the limited data, response to neutron irradiation was roughly the same as most of metals, which showed increase in strength (irradiation hardening), decrease in ductility, and increase in DBTT [25, 110, 111, 113–115].

Krautwasser *et al*. [25] reported the effects of neutron irradiation at low temperature (252–302°C) on DBTT of pure W and W-10%Re alloy. Before irradiation, W-10%Re alloy showed a lower DBTT compared to the pure W, which is the same tendency as the present study. Up to approximately 2×10^{24} m^{-2} in fast neutron fluence, DBTT of W-10%Re was lower than that of pure W. However, a more rapid increase in DBTT of W-10%Re by neutron irradiation than that in pure W was found above this neutron irradiation dose. Thus, positive and negative effects of Re-addition, which were dependent on the irradiation dose, were also indicated in the macroscopic mechanical properties, as well as the irradiation hardening, although irradiation temperature of this DBTT study was relatively low, compared to the studies on irradiation hardening.

Recent collaboration project by Japan and U.S.A., called PHENIX (2013–2018), carried out high temperature neutron irradiation tests of the pure and modified W materials developed in the present study in HFIR using thermal neutron shielding [116]. As shown in Fig. 27 [110, 111], K-doping and Re-addition showed a positive effect on the suppression of ductility loss. Especially, materials alloyed by Re (W-3%Re (H) and K-doped W-3%Re (H) plates) showed a finite value of elongation, even at 500°C. In the non-irradiated condition, these two materials showed higher UE compared to the non-alloyed materials, as shown in Fig. 16. Thus, it is possible that the ability of work hardening of these materials could be maintained even after the neutron irradiation. As another positive effect, increase in $DBTT_{Tensile}$ by neutron irradiation was also suppressed by K-doping and Re-addition. Increases in $DBTT_{Tensile}$ by neutron irradiation of pure W (H), K-doped W (H), W-3%Re (H), and K-doped W-3%Re (H) plates were approximately 600°C, 500°C, below 400°C, and below 400°C, respectively. Further neutron irradiation studies up to higher doses at various temperatures are expected to clarify the effectiveness of modification by K-doping, dispersion of La_2O_3 particles, and Re-addition.

6.3.3. Effects of transmutant helium

Higher energy neutrons, e.g., 14 MeV neutrons in fusion reactor, could cause gas transmutation via (n, α) reaction, resulting in the accumulation of helium. Basically, the number of research on helium effects in bulk W materials are limited compared to that on the effects of high-concentration helium related to plasma-surface interaction of PFMs. Chernikov *et al*. [117] reported microstructural evolutions of pure W after helium implantation at 52°C up to 600 appm and post-implantation annealing at a maximum temperature of 2100°C. Formation of helium nano-bubbles in W started after annealing above 1350°C, however, no significant swelling and no segregation of helium to grain boundaries were observed, which might result in the grain boundary embrittlement. In contrast, the concentration of helium produced by transmutation in W-based PFMs of divertors of DEMO reactor was calculated to below 20 appm after 5 years' operation [118]. Therefore, effects of low concentration helium are more important. Hasegawa *et al*. [119] reported Vickers hardness change of pure W (H) plate after helium implantation below 100°C up to 20 appm

and post-implantation isochronal annealing at 1100–1500°C for 1 h. Helium implantation tests were carried out using a cyclotron accelerator of Tohoku university. Specimens were implanted by 50 MeV α–particle beam of the cyclotron accelerator. An energy degrader was used to obtain a uniform helium distribution along the specimen thickness. As shown in Fig. 28 (b), only 20 appm helium suppressed the recrystallization of pure W (H) plate. Based on the analyses of grain structure and microstructure, this could be attributed to no change of dislocation cell structure and sub-grains by helium atoms and bubbles. A TEM bright field image shown in Fig. 28 (a) could indicate the suppression of dislocation movement by helium. Due to this suppression of recrystallization by a low concentration helium, no change of tensile properties was observed [120]. Although the pure W (H) plate with no helium implantation showed decrease in strength and increase in elongation after annealing at 1500°C for 1 h, as shown in Fig. 19, the same material after low concentration helium implantation showed a similar stress-strain curve to that of the as-received one (non-implanted and non-annealed). These results could indicate a positive effect of transmutant helium as an increase in recrystallization temperature.

6.3.4. Integrated thermo-mechanical properties under heat load environments

As mentioned in section 1, PFMs of fusion reactor divertor must survive the complicated heat loads, including steady state, transient, and ELM heat loads. Under these heat load environments, integrated thermo-mechanical properties, as well as individual properties (tensile, creep, and fatigue, etc.) evaluated by material tests, are important for the PFMs because various types of mechanical and thermal loadings would be simultaneously applied. To evaluate the effects of such heat loads in fusion reactor, simulation experiments have been performed using various types of test facilities, e.g., ion beam test facilities like GLADIS [121], plasma gun facilities like QSPA Kh-50 [122], linear plasma generators like MAGNUM-PSI [123], and electron beam test facilities like JUDITH 1 [124], JUDITH 2 [125], ACT2 [126], and JEBIS [127].

To clarify the effectiveness of modifications by K-doping and Re-addition under such heat load environments, thermal shock tests of pure and modified W materials using JUDITH 1 under ELM-like heat load were conducted as an attempt in the present study [128]. As shown in the surface images of Fig. 29, pure and all modified materials showed a high number density of micro-cracks on heat-loaded surfaces. However, the number and depth of micro-cracks of modified materials, except K-doped W (L) rod, were smaller compared to the pure W (H) plate, as shown in the histograms of Fig. 29. Especially, W-3%Re (H) plate showed relatively shallow micro-cracks with low number density. In contrast, small number of deep and long macro-cracks was observed only in the pure W (H) and K-doped W-3%Re (H) plates, which were formed along direction of L × T surfaces. K-doped W-3%Re (H) plate showed excellent impact properties along L-S direction, no negative tensile properties along L, T, and S directions, and no negative fatigue properties along L direction, compared to the other modified materials, as mentioned in sections 4, 5, and 6.1. Therefore, further investigations are planned as future work to clarify the effectiveness of K-doped W-3%Re (H) plate, including e.g., investigations of impact, fatigue, and the other mechanical properties especially along S direction and effects of stress triaxiality under thermal shock tests, which could change plastic deformability of materials [129].

According to this thermal shock test campaign, individual effectiveness of K-doping and Re-addition was indicated, however, effectiveness of simultaneous application of K-doping and Re-addition was not clear. In contrast, a finite element analysis, which simulated cyclic high heat flux tests of ITER-like monoblock divertor using modified W materials as a PFM, indicated a possibility of the extension of fatigue life of a divertor using both K-doped W (H) and K-doped W-3%Re (H) plates [130, 131]. Therefore, further evaluations of integrated thermo-mechanical properties have to be performed to clarify their effectiveness under actual fusion reactor environments.

7. Summary and outlook

The objective of this paper was to summarize current understanding and issues related to the thermo-mechanical properties of W materials developed under collaboration R&D by universities in Japan for the last decade, which were aimed to achieve materials with improved thermo-mechanical properties, neutron irradiation tolerance, and the possibility of mass-production with microstructural uniformity for PFMs of future fusion reactor divertors.

In this paper, the effects of grain refining, K-doping, dispersion strengthening by La_2O_3 particles, and alloying by Re were discussed in sections 3, 4, and 5 from the viewpoints of high-temperature microstructural stability, Charpy impact properties, and tensile properties. In addition, an assessment of long-term structural reliability and lifetime is essential in future fusion reactors like

DEMO. Therefore, fatigue life, long-term microstructural stability, and effects of neutron irradiation of pure and modified W materials developed in this R&D were also discussed in section 6. Furthermore, PFMs of fusion reactor divertor will be exposed to complicated heat loads, including steady state, transient, and ELM heat loads. Under these heat load environments, integrated thermo-mechanical properties are important because various types of loadings would be simultaneously applied. Therefore, the possibility of improving the structural strength and lifetime of divertors by using the modified materials as PFMs were also discussed in section 7 based on the results of heat load tests and structural analysis. The present study yielded several results that are summarized as follows:

7.1. Grain structure and hardness of as-received materials

Grain size of materials with the same major chemical composition decreased with increasing deformation ratio. As for the materials fabricated under the same deformation ratio, grain size decreased by K-doping, dispersion of La_2O_3 particles, and Re-addition. Re-addition caused solid solution softening, whereas K-doping and dispersion of La_2O_3 particles increased hardness.

7.2. High-temperature microstructural stability

Recrystallization was suppressed by K-doping, dispersion of La_2O_3 particles, and Re-addition. The materials with higher deformation ratio showed slightly lower recrystallization temperature, compared to those with lower deformation ratio with the same major chemical compositions. The positive effects of K-doping and Re-addition were also observed even during long-term annealing. Especially, materials alloyed by Re showed no recrystallization after annealing at 1100°C for 3000 h.

7.3. Grain growth during annealing

A significant grain growth of pure W occurred during the primary and the secondary recrystallizations. In contrast, K-doping, dispersion of La_2O_3 particles, and Re-addition had an ability to suppress grain growth during both stages. Especially for grain growth caused by the secondary recrystallization at relatively high temperature, K-doping and a dispersion of La_2O_3 particles were more effective than Re-addition.

7.4. Strength

Strengthening by K-doping, dispersion of La_2O_3 particles, and Re-addition were observed, while the effect of difference in deformation ratio was small. Anisotropy of tensile strength was observed regardless of materials, especially at low temperatures. Decrease in strength occurred by recrystallization.

7.5. Ductility

Positive effects of K-doping, dispersion of La_2O_3 particles, and Re-addition on ductility by tensile tests were limited. The materials alloyed by Re showed higher uniform elongation and lower reduction in area. Anisotropy of elongation was observed regardless of materials, especially at low temperatures. Significant increase in elongation occurred by recrystallization.

7.6. DBTT by tensile test

Approximately 50–100°C reduction in $DBTT_{Tensile}$ was caused by K-doping, dispersion of La_2O_3 particles, and Re-addition. Increase in $DBTT_{Tensile}$ by recrystallization was roughly 100−300°C. $DBTT_{Tensile}$ increased with increase in strain rate.

7.7. DBTT by Charpy impact test

As for the high-deformed materials, approximately 200 and 100°C reduction in $DBTT_{Charpy}$ and approximately 40 and 30% increase in USE were caused by the K-doping and Re-addition, respectively. Moreover, synergistic effects of K-doping and

Re-addition were clearly observed. In contrast, low deformation ratio produced very low absorbed energy. No significant positive effects by dispersion of La_2O_3 particles were observed in the low-deformed materials. The effect of dispersion of La_2O_3 particles has to be clarified in future work by applying to the high-deformed materials. $DBTT_{Charpy}$ was decreased by grain refining, where Hall–Petch-type relations between $DBTT_{Charpy}$, USE, and grain size were observed. Recrystallization could produce brittle intergranular fracture with zero absorbed energy, even at 1000°C. Anisotropy of DBTT have to be investigated as a future work.

8. Fatigue life

Effect of test temperature difference ranging from 500 to 1232°C on fatigue life seemed to be small in pure W materials. Modified W materials showed no significant improvement compared to pure W. Recrystallization produced slightly short fatigue life at higher temperature and significantly short fatigue life at room temperature. Further evaluations at widely-ranged test temperatures have to be performed.

8.1. Neutron irradiation hardening

Irradiation hardening of pure W at temperatures up to around 800°C increased with displacement damage up to approximately 0.5 dpa, which could be mainly due to void formation, and saturated above this dose range, if solid transmutation by thermal neutron could be negligible. The addition of a few percent of Re caused suppression of irradiation hardening due to suppression of void formation below approximately 0.5 dpa, whereas no suppression of irradiation hardening was observed above this dose range because precipitations could start to form in the W-Re alloys. Up to around 0.5 dpa, no clear effects of K-doping and dispersion of La_2O_3 particles on irradiation hardening and microstructure development were found.

8.2. Macroscopic mechanical properties after neutron irradiation

Limited investigations of macroscopic mechanical properties, especially under higher doses at various temperatures, are significant issue for future fusion reactor development. According to the recent irradiation study in PHENIX project by Japan and U.S.A., K-doping and Re-addition showed a positive effect on the suppression of ductility loss. Increase in $DBTT_{Tensile}$ by neutron irradiation was also suppressed by K-doping and Re-addition.

8.3. Effects of transmutant helium

Higher energy neutrons, e.g., 14 MeV neutrons in fusion reactor, could cause gas transmutations via (n, α) reaction, resulting in accumulation of helium. Concentration of helium produced by transmutation in W-based PFMs of divertors of DEMO reactor was calculated to below 20 appm after 5 years' operation. A recent study using accelerator-based helium implantation revealed a suppression of recrystallization of pure W by only 20 appm helium. Further studies have to be performed to clarify the detailed effects of transmutant helium on pure and modified W materials.

8.4. Integrated thermo-mechanical properties under heat load environments

According to the thermal shock tests under ELM-like heat load, individual effectiveness of K-doping and Re-addition was indicated, however, effectiveness of simultaneous application of K-doping and Re-addition was not clear. In contrast, a finite element analysis indicated possibility of extension of fatigue life of divertor using both K-doped W and K-doped W-3%Re. Further evaluations of integrated thermo-mechanical properties have to be performed to clarify their effectiveness under actual fusion reactor environments.

As mentioned in section 1, the upper and lower limits of operation temperature of divertor components using W material would be determined by the recrystallization temperature and DBTT, respectively. Fig. 30 shows the $DBTT_{Tensile}$, $DBTT_{Charpy}$, and recrystallization temperature by isochronal annealing for 1 h (T_{Rec}) of materials investigated in the present study. Based on these indexes, K-doped W-3%Re (H) plate could be a better solution for PFM of fusion reactor divertors. It was clarified that strength, ductility, and low cycle fatigue life of this material was comparable to the other materials, long-term microstructural stability of this material was better than the other materials, and effects of complicated heat loads under actual fusion reactor environments might not be serious based on investigations of the present study. Moreover, if considering the neutron irradiation up to approximately 0.5 dpa, this material could show no significant degradation. However, materials alloyed by Re have an intrinsic concern of higher irradiation hardening caused by neutron irradiation up to higher doses, which could be mainly caused by the irradiation-induced precipitation. As one of the concerns related to macroscopic mechanical properties, it was reported that higher DBTT was produced in W-Re binary alloys compared to pure W after high dose irradiation. In addition, it is possible that the solid transmutation could enhance these issues. Therefore, investigations of thermo-mechanical properties, especially under higher dose neutron irradiation at various temperatures, are significantly necessary for future fusion reactors to clarify the effectiveness of modified W materials in the present study under long-term operation. On the other hand, it would be desirable that the complete solid solution W-X alloy systems (X = tantalum (Ta), molybdenum (Mo), niobium (Nb), and vanadium (V), etc.) are additionally considered as an alternative alloying element to avoid possibility of irradiation-induced precipitation. In contrast, K-doping showed several positive effects and no negative effects on both short- and long-term material properties and phenomena within the present and previous studies (effects of dispersion of La_2O_3 particles are not fully clarified at present.). Therefore, the second-phase dispersion strengthening should be considered as one of the effective modification methods.

Conflicts of Interest Statement

None

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Fig. 1. Schematic illustrations of (upper) heat loads, neutron and ions irradiations, and temperature of monoblock divertor using a W material, and metallographic structures of pure W in as-received condition and after isochronal annealing at 1100–1800°C for 1 h, which is fabricated by powder metallurgy and hot-rolling with stress-relief heat treatment in the present study and (lower) surface temperature change of divertor due to stationary and transient heat loads during operation, where the maximum surface temperature was (left) between DBTT and recrystallization temperatures and (right) above recrystallization temperature.

Fig. 2. Images of (a) K-bubbles and (b) La_2O_3 particles in W observed by ultra-high voltage transmission electron microscopy (UHVEM) and scanning transmission electron microscopy (STEM).

Fig. 3. 3D inverse pole figure (IPF) images obtained by electron backscatter diffraction (EBSD) and 3D metallographic images obtained by optical microscope of (a) pure W (H) and (b) K-doped W-3%Re (H) plates in as-received condition [47–50] and (c) transmission electron microscopy (TEM) and IPF images of pure W (H) plate in as-received condition.

Fig. 4. Metallographic images obtained by optical microscope of (a) pure W (H), (b) pure W (L), (c) K-doped W (H), and (d) K-doped W (L) swaged rods in as-received condition.

Fig. 5. Thermal conductivity of pure W (H), K-doped W (H), W-1%Re (H), W-3%Re (H), K-doped W-3%Re (H) plates, and K-doped W (L) rod [53, 54].

Fig. 6 Scanning electron microscope (SEM) image of fracture surface of (a) specimen made of as-received K-doped W (L) rod after tensile test along radial direction at 400°C and (b) specimen made of recrystallized K-doped W-3%Re (H) plate after Charpy impact test at 600°C.

Fig. 7. Annealing temperature dependences of Vickers hardness of (a) pure W (H), K-doped W (H), W-3%Re (H), and K-doped W-3%Re (H) plates, (c) pure W (H), W-3%Re (L), K-doped W-3%Re (L), and W-3%Re-1%La_2O_3 (L) plates, and (d) pure W (H), K-doped W (H), pure W (L), and K-doped W (L) rods. KAM images obtained by EBSD of as-received and annealed pure W (H), K-doped W (H), and K-doped W-3%Re (H) plates are shown in (b). Measured surface and annealing time were L × S surface and 1 h, respectively (part of data were from [50]).

Fig. 8. (a). Annealing temperature dependences of grain size along S direction (d_S) of pure W (H), K-doped W (H), W-3%Re (H and L), and K-doped W-3%Re (H and L), and W-3%Re-1%La_2O_3 (L) plates. IPF images obtained by EBSD of annealed pure W (H), K-doped W (H), and K-doped W-3%Re (H) plates are shown in (b). Measured surface and annealing time were L × S surface and 1 h, respectively (part of data were from [50]).

Fig. 9. Test temperature dependences of absorbed energy of Charpy impact tests of KLST specimens (L-S direction) made of as-received pure W (H) plate in the present study, as-received pure W plate (t4 mm), and as-received pure W round-blank (φ175 mm x t29 mm). Appearances of tested specimens of pure W (H) plate are shown on the right side [47, 51, 58–62].

Fig 10. Test temperature dependences of absorbed energy of Charpy impact tests of KLST specimens (L-R direction) made of as-received K-doped W (H, I, and L) rods. Appearances of tested specimens of K-doped W (H and L) rods are shown on the right side.

Fig. 11. Test temperature dependences of absorbed energy of Charpy impact tests of KLST specimens (L-S direction) made of as-received pure W (H), K-doped W (H), W-3%Re (H and L), K-doped W-3%Re (H), and W-3%Re-1%La_2O_3 (L) plates. Appearances of tested specimens of W-3%Re (H and L) plate are shown on the right side (part of data were from [47–50]).

Fig 12. Relationships between grain size along thickness (d_S) and USE and DBTT obtained by Charpy impact tests of KLST specimens (L-S direction) of as-received pure W (H), K-doped W (H), W-3%Re (H), and K-doped W-3%Re (H) plates. Data of as-received pure W plate (t4 mm), and as-received pure W round-blank (φ175 mm x t29 mm) are also plotted [47–51, 58–62].

Fig. 13. Fracture surfaces of specimens after Charpy impact tests of (a) as-received pure W (H) plate tested at 400°C and (b) as-received K-doped W-3%Re (H) plate tested at 200°C [48, 50].

Fig 14. Test temperature dependences of absorbed energy of Charpy impact tests of KLST specimens (L-S direction) made of pure W (H), K-doped W (H), K-doped W-3%Re (H), and W-3%Re-1%La_2O_3 (L) plates before and after the isochronal annealing at (a) 1100, (b) 1400, and (c) 2300°C for 1 h. Solid and open symbols correspond to data before and after the annealing, respectively.

Fig. 15. Fracture surfaces of specimens after Charpy impact tests of (a) pure W (H) plate annealed at 2300°C and tested at 600°C and (b) K-doped W-3%Re (H) plate annealed at 2300°C and tested at 600°C.

Fig. 16. Test temperature dependences of (a, b) ultimate tensile strength (UTS), (c, d) total elongation (TE), (e, f) uniform elongation (UE), and (g, h) reduction in area (RA) by tensile tests (strain rate = 1×10^{-3} s^{-1}) of SS-J specimens (L direction) of pure W (H), K-doped W (H), W-3%Re (H and L), K-doped W-3%Re (H and L), and W-3%Re-1%La_2O_3 (L) plates in as-received condition (part of data were from [48–50]).

Fig. 17. Test temperature dependences of (a) ultimate tensile strength (UTS) and (b) total elongation (TE) by tensile tests (strain rate = 1×10^{-3} s^{-1}) of SS-J specimens (L direction) of K-doped W (H, I, and L) rods in as-received condition (part of data were from [52, 67]).

Fig. 18. Test temperature dependences of ultimate tensile strength (UTS) and total elongation (TE) by tensile tests (strain rate = 1×10^{-3} s^{-1}) of SS-J specimens (L, T, and R direction) and VS-T specimens (S direction) of (a, d) pure W (H) plate, (b, e) K-doped W (H) plate, (c, f) K-doped W-3%Re (L) plate, (g, i) pure W (L) rod, and (h, j) K-doped W (L) rod in as-received condition [67, 68].

Fig. 19. Test temperature dependences of ultimate tensile strength (UTS) and total elongation (TE) by tensile tests (strain rate = 1×10^{-3} s^{-1}) of SS-J specimens (T direction) of (a, b) pure W (H), (c, d) K-doped W (H), (e, f) K-doped W-3%Re (H), and (g, h) K-doped W-3%Re (L) plates before and after the isochronal annealing at 1500 and 2300°C for 1 h.

Fig. 20. Strain rate dependences of 0.2% proof stress ($\sigma_{0.2}$) by tensile tests of SS-J specimens (L direction) at room temperature, 100, 200, 300, 400, 500, and 700°C of pure W (H) plate in as-received condition [73].

Fig. 21. Test temperature dependences of strain sensitivity (m) evaluated using 0.2% proof stress ($\sigma_{0.2}$) by tensile tests of SS-J specimens (L direction) at room temperature, 100, 200, 300, 400, 500, and 700°C of pure W (H), K-doped W (H), W-3%Re (H), K-doped W-3%Re (H) plates and K-doped W (H and L) rods in as-received condition (part of data were from [73, 75]).

Fig. 22. Strain rate dependences of total elongation (TE) by tensile tests of SS-J specimens (L direction) at room temperature, 100, 200, and 300°C of pure W (H) plate in as-received condition [73].

Fig. 23. Relationship between total strain range and number of cycles to failure of pure W (H) plate, K-doped W (H) plate, K-doped W-3%Re (H) plate, and K-doped W (H) rod along L direction in as-received and annealed conditions. Data of commercial grade pure W plate (6 mm thickness), commercial grade pure W rod (6 mm diameter), and pure W plate (14 mm thickness) reported by Schmunk *et al*. [77, 78] are also plotted. "T_{anneal}" and "T_{test}" in this figure indicate the annealing and test temperatures, respectively. The arrow in the blue, square plot indicates that the fatigue life was not reached, but the test was stopped at that number of cycles. Fracture surfaces (#1, #2, and #3) of pure W plates are also shown.

Fig. 24. (a). Annealing time dependences of Vickers hardness of pure W (H), K-doped W (H), W-3%Re (H), and K-doped W-3%Re (H) plates. IPF images obtained by EBSD of as-received and annealed pure W (H), K-doped W (H), and K-doped W-3%Re (H) plates are shown in (b). Measured surface and annealing temperature were L × S surface and 1100°C, respectively.

Fig. 25. Relationships between irradiation hardening evaluated by Vickers hardness and displacement damage of (a) pure W [43, 94, 95, 97–99, 103], (b) W-Re binary alloys [43, 94, 95, 97–99], and (c) dispersion-strengthened W materials [100, 102, 110, 111] after neutron irradiation in Joyo (fast breeder

reactor), JMTR (mixed-spectrum fission reactor), and HFIR (mixed-spectrum fission reactor). Legends in these graphs indicate "material (conditions of fabrication and heat-treatment) / reactor for neutron irradiation / range of irradiation temperature." R, AC, SX, and SR indicate materials after recrystallization heat treatment, materials fabricated by arc-melting, single-crystal materials, and materials after stress-relief heat treatment, respectively. HFIR (NS) and HFIR (S) indicate HFIR with no thermal neutron shielding and HFIR with thermal neutron shielding, respectively.

Fig. 26. Relationships between nano-indentation hardness and displacement damage of pure W (H), K-doped W (H), W-3%Re (H), K-doped W-3%Re (H), and W-3%Re-1%La_2O_3 (L) plates and K-doped W rod (L) after irradiation by W-ion and proton. R and SR in legends mean materials after recrystallization heat treatment and stress-relief heat treatment, respectively (part of data were from [54, 112]).

Fig. 27. Test temperature dependences of (a) ultimate tensile strength (UTS) and (b) total elongation (TE) by tensile tests (strain rate = 1×10^{-3} s^{-1}) of SS-J specimens (L direction) of pure W (H), K-doped W (H), W-3%Re (H), and K-doped W-3%Re (H) plates before and after neutron irradiation. Combinations of displacement damage / irradiation temperature / test temperature were 0.4 dpa / 600°C / 500°C, 0.7 dpa / 800°C / 700°C, and 0.7 dpa / 1000°C / 900°C, respectively. Stress-strain curves of tensile tests at 500°C before and after the irradiation at 600°C up to 0.4 dpa are shown in (c) [110, 111].

Fig. 28. (a). TEM bright field images of pure W (H) plate after helium implantation and annealed at 1500°C and (b) annealing temperature dependences of Vickers hardness of pure W (H) plate before and after helium implantation. Concentration of implanted helium, implantation temperature, measured surface, and annealing time were 20 appm, below 100°C, L × T surface, and 1 h, respectively [119].

Fig. 29. Surface images obtained by SEM and histograms of crack depth distribution obtained by cross-sectional optical microscope observation of as-received pure W (H) plate, K-doped W (H) plate, W-3%Re (H) plate, K-doped W-3%Re (H) plate, and K-doped W (L) rod after thermal shock tests at 0.38 GW/m^2 on T × S surfaces of plates and radial surface of rod. The yellow arrows indicate the macro-cracks [128].

Fig. 30. Summary of DBTT obtained by total elongation of tensile tests ($DBTT_{Tensile}$ from table 4), DBTT obtained by Charpy impact tests ($DBTT_{Charpy}$ from table 3), and recrystallization temperature by isochronal annealing for 1 h (T_{Rec} from table 2) of pure W (H), K-doped W (H), W-3%Re (H and L), K-doped W-3%Re (H and L), and W-3%Re-1%La_2O_3 (L) plates and pure W (H and L) and K-doped W (H, I, and L) rods in the present study.

Table 1. Grain size along L, T, and S directions (d_L, d_T, and d_S) of pure W (H) plate, K-doped W (H) plate, W-3%Re (H and L) plates, K-doped W-3%Re (H and L) plates, W-3%Re-1%La_2O_3 (L) plates and grain sizes along L and R directions of pure W (H and L) rods and K-doped W (H and L) rods in as-received condition (part of data were from [47–50, 52]).

Material	Deformation Ratio	Grain size [μm]				
		d_L	d_T	d_S	d_R	
Plate	Pure W	H	98	59	22	–
	K-doped W	H	30	20	11	–
	W-3%Re	H	52	39	19	–
		L	61	35	28	–
	K-doped W-3%Re	H	33	20	8	–
		L	35	29	21	–
	W-3%Re-1%La_2O_3	L	23	17	12	–
Rod	Pure W	H	121	–	–	7.3
		L	111	–	–	58
	K-doped W	H	105	–	–	5.7
		L	111	–	–	55

Table 2. Recrystallization temperature of pure W (H) plate, K-doped W (H) plate, W-3%Re (H and L) plates, K-doped W-3%Re (H and L) plates, W-3%Re-1%La_2O_3 (L) plates, pure W (H and L) rods and K-doped W (H and L) rods based on the Vickers hardness after isochronal annealing for 1 h.

Material	Deformation Ratio		Recrystallization temperature [°C]
Plate	Pure W	H	1250
	K-doped W	H	1350
	W-3%Re	H	1500
		L	1400
	K-doped W-3%Re	H	1450
		L	1550
	W-3%Re-1%La_2O_3	L	1550
Rod	Pure W	H	1400

		L	1700
	K-doped W	H	1600
		L	1700

Table 3. DBTT obtained by Charpy impact tests of pure W (H), K-doped W (H), W-3%Re (H and L), K-doped W-3%Re (H), and W-3%Re-1%La_2O_3 (L) plates (L-S direction) and K-doped W (H, I, and L) rods (L-R direction) in as-received condition.

Material	Deformation Ratio	Test direction	DBTT [°C]	
Plate	Pure W	H	L-S	550
	K-doped W	H		350
	W-3%Re	H		450
		L		550
	K-doped W-3%Re	H		250
	W-3%Re-1%La_2O_3	L		550
Rod	K-doped W	H	L-R	300
		I		350
		L		650

Table 4. $DBTT_{Tensile}$ obtained by total elongation of tensile tests of pure W (H), K-doped W (H), W-3%Re (H and L), K-doped W-3%Re (H), and W-3%Re-1%La_2O_3 (L) plates (L direction) and Pure W (L) and K-doped W (H, I, and L) rods (L direction) in as-received condition.

Material	Deformation Ratio	Test direction	$DBTT_{Tensile}$ [°C]	
Plate	Pure W	H	L.D.	150
	K-doped W	H		50
	W-3%Re	H		100
		L		100
	K-doped W-3%Re	H		<R.T.
		L		50
	W-3%Re-1%La_2O_3	L		50

Rod	Pure W	L	L.D.	300
	K-doped W	H		<200
		I		<200
		L		<200