Effect of potassium bubbles on the thermal shock fatigue behavior of large-volume potassium-doped tungsten alloy

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Received 3 August 2022, revised 10 October 2022
Accepted for publication 14 October 2022
Published 10 November 2022

Abstract
A newly developed large-volume potassium-doped tungsten (W–K) plate with a thickness of 15 mm and a weight of 25 kg by powder metallurgy plus hot rolling was prepared to meet the requirements of the International Thermonuclear Experimental Reactor (ITER) in engineering application. In order to clarify the effect of K doping on the thermal shock performance of W–K alloy, transient thermal shock tests with a single-pulse duration of 1 ms for 100 shots at room temperature were performed. The absorbed power density is set to 0.33, 0.44, 0.55 and 0.66 GW m⁻², respectively. Furthermore, the microstructure, Vickers micro-hardness before and after the transient thermal shock, thermal conductivity and relative density were also characterized. The results indicate that the cracking threshold of rolled W–K is 0.44–0.55 GW m⁻², which possesses a better transient thermal shock resistance compared with the most of advanced W-based materials. This is mainly because K doping can significantly improve the high-temperature stability and mechanical properties of W material without reducing its thermal conductivity. In particular, K bubbles can also effectively inhibit the formation and propagation of cracks during thermal shock. Moreover, the cracking mechanism of rolled W–K alloy is also discussed in detail. This study is helpful for building a trusted ITER database on advanced W-based materials that provides useful references for the selection of future plasma-facing materials.

Keywords: potassium-doped tungsten alloy, potassium bubble strings, transient thermal shock, microstructure, formation and propagation of cracks

(Some figures may appear in colour only in the online journal)
1. Introduction

Plasma-facing materials (PFMs) will inevitably suffer from extremely harsh conditions during the operation of ITER and future fusion reactors, such as transient- and steady-state thermal loads, plasma irradiation, neutron irradiation, neutral atom and electron bombardment [1], etc. Among them, transient-state thermal-load edge localized models (ELMs) can generate strong heat fluxes of up to ~GW m$^{-2}$ to deposit on the PFMs within milliseconds. This will cause serious PFM damage, such as surface melting, cracking and roughening [2], strongly deteriorating the thermal properties of the PFM and shortening its service life.

Pure tungsten (PW) has been considered as one of the most potential PFMs in future fusion armor due to its superior mechanical properties, high melting point, low hydrogen isotope retention, excellent thermal conductivity [3, 4], etc. Nevertheless, some shortcomings of PW, including recrystallization embrittlement, low-temperature brittleness (high ductile–brittle transition temperature, DBTT ~ 400 °C) and irradiation-caused embrittlement, severely limit its application in the domain of nuclear energy [5, 6]. It is known that the mechanical properties of materials, especially low-temperature brittleness, are key factors affecting its thermal shock resistance. In general, the formation of surface cracks under thermal loads can be primarily attributed to the heat stress caused by the temperature gradient formed during heating and cooling [7]. The conditions of crack formation are concluded to be the following (i) when (the DBTT of PFMs) < (surface temperature) during thermal loading, (ultimate tensile strength of PFMs) < (maximum thermal stress); (ii) when (the DBTT of PFMs) > (surface temperature) during thermal loading, (yield strength of PFMs) < (maximum thermal stress). Therefore, improving the tensile strength and decreasing the DBTT of PFMs are effective methods to improve the thermal shock resistance.

For decades, researchers have tried many methods to improve the mechanical performance of PW, such as equal-channel angular pressing (ECAP) [8], high-pressure torsion (HPT) [9], rolling [10], forging, etc [11]. Nevertheless, the thermostability and low-temperature brittleness still are inadequate to meet the demands of fusion reactors. Hence, all kinds of W-based alloys were developed to improve the tensile strength and decrease the DBTT, such as carbides [12, 13], oxides [14, 15], dispersion strengthened W, W-vanadium [16], W-rhenium [17], potassium-doped W (W–K) alloys [18, 19], etc. Among them, W–K alloys have been deemed to be one of the most potential PFMs because it can simultaneously improve the mechanical properties [20] and neutron irradiation resistance [21]. For example, the mechanical and neutron irradiation properties of W–K alloys have been extensively studied by Fukuda et al [22, 23] and Nogami et al [24–27]. The results showed that K doping can effectively improve the mechanical and irradiation properties of W materials. In addition, Fukuda et al [28] indicated that W–K alloys possess almost the same thermal conductivity as pure W. Furthermore, W–K also has excellent recrystallization and high-temperature creep resistance, which is primarily attributed to the fact that K bubbles as a second phase can play a role in pinning the grain boundaries and dislocations [29]. Consequently, the thermal shock resistance of W–K has aroused increasing interest.

In recent years, the transient thermal shock behavior of small-sized W–K rods and plates has been widely studied. For example, Tang et al [30] conducted a systematic study on PW and W–K prepared via spark plasma sintering (SPS) technology, and the transient-state thermal shock resistance of SPS-PW and SPS-82KW at room temperature (RT) with a single-pulse duration of 5 ms for 100 cycles was compared. The results showed that SPS-82WK presents a higher cracking threshold of 0.37–0.50 GW m$^{-2}$ compared to SPS-PW (<0.37 GW m$^{-2}$) [30]. Pintsuk et al [31] fabricated W–K rods with a diameter of 15 mm by a forging process and investigated the transient-state thermal shock resistance at various base temperatures up to 600 °C with a single-pulse duration of 1 ms for 100 cycles. The results indicated that the cracking threshold is higher than 0.95 GW m$^{-2}$ [31]. Previously, our group also prepared a W–K rod with a diameter of 9 mm by rolling plus swaging, and its cracking threshold is in the range of 0.44–0.66 GW m$^{-2}$ after transient-state thermal shock at RT with a single-pulse duration of 5 ms for 1 cycle [32]. Nevertheless, so far, the service performance of large-volume W–K plate that meets the requirements of engineering applications is still little known because of the extraordinary difficulty in machining. Therefore, our group fabricated a newly developed large-volume W–K plate with a thickness of 15 mm and a weight of 25 kg by powder metallurgy plus hot rolling, which meets the requirements of the International Thermonuclear Experimental Reactor (ITER) in engineering application, i.e. the thickness of the W–Cu mono-block as a divertor component is not less than 12 mm [33]. Furthermore, some of its key properties have been characterized and the results indicate that the W–K exhibits high strength and large plasticity simultaneously, superior high-temperature stability [34], good plasma exposure resistance [35] and excellent irradiation hardening resistance [36]. In order to more systematically evaluate the service performance of this W–K plate, it is very important and necessary to investigate its thermal shock resistance.

In this work, the transient-state thermal shock behavior of W–K was appraised and the results indicate that W–K presents an excellent thermal shock resistance. In addition, the thermal conductivity, microstructure and micro-hardness before and after thermal shock were characterized. Most importantly, the influence of K bubbles on the formation and propagation of cracks during thermal shock is discussed in detail. These studies are beneficial for building a trusted ITER database on advanced W-based materials that is able to provide a useful reference for numerical simulation under high heat flux and the selection of future PFMs.
2. Experimental

2.1. Preparation of hot-rolled W–K plate

Tungsten trioxide powder was doped into a mixed solution of potassium silicate and aluminum nitrate via chemical doping and then dried. The uniformly mixed powder was subsequently subjected to hydrogen reduction, acid washing and drying in sequence. In this way, so-called AKS-doped W powder was obtained. Afterwards, a large-volume W–K plate with a rolling reduction of 78% and a weight of 25 kg was prepared via pressing, sintering, hot rolling and annealing. The length, width and thickness of the obtained plate were 460, 190 and 15 mm, respectively. The rolling, transverse and normal direction are recorded as RD, TD and ND, respectively. The impurity composition, nature of the K bubbles, detailed fabrication procedure and digital photo of the W–K plate were presented in [34]. Subsequently, samples with a size of $10 \times 10 \times 1.5$ mm³ were obtained via electrical discharge machining and mechanical polishing. Finally, all samples were annealed at $800 \degree C$ for 1 h in a vacuum furnace to eliminate residual stress caused via mechanical polishing. A flow diagram of the experimental process is displayed in figure 1.

2.2. Experimental procedures

Thermal conductivity ($\lambda$), an important parameter determining the thermal shock performance of materials, can be calculated via equation (1):

$$\lambda = \alpha \cdot C_p \cdot \rho,$$

where $C_p$ and $\alpha$ are the specific heat ($J \, kg^{-1} \, K^{-1}$) and thermal diffusivity, respectively, which were measured using thermal analyzing apparatus (NETZSCH STA449 F3) and laser flash diffusivity technology (NETZSCH LFA467). The Archimedes method was applied to determine the relative density ($\rho$) of materials, $\sim 99.5\%$.

High thermal load tests were carried out on the EMS-60 facility at the Southwestern Institute of Physics (China), which can provide a maximum electron beam power of 60 kW. The electron beam was loaded on the RD–TD surface and the dimensions of the loading area were $3.4 \times 3.4$ mm². Detailed parameters of EMS-60 can be found in [37]. Figure 2 presents a schematic drawing of the sampling and thermal loading direction of thermal shock samples. According to our previous work [32], the cracking threshold of rolled plus swaged W–K was evaluated to be $>0.44 \, GW \, m^{-2}$ under 100 shots with a single-pulse duration of 1 ms at RT. Consequently, in this work, absorbed power densities (APDs) of 0.33, 0.44, 0.55 and 0.66 GW m⁻² were set to study the cracking regime of rolled W–K at RT with a single-pulse duration of 1 ms for 100 shots. The pulse interval was selected to be 5 s to guarantee that the surface temperature of the specimen could be cooled to RT after each individual pulse. The maximum surface temperature $T$ during thermal loading can be estimated by equation (2):

$$T = T_0 + 2P \sqrt{\frac{t}{\pi \lambda \rho C_p}},$$

in which $T_0$ is RT; $t$ and $P$ are the shot duration (s) and APD, respectively. In order to ensure the accuracy of the experimental results, three repeated samples for each loading condition were performed. In addition, the Vickers micro-hardness test with an invariant load of 200 g for 15 s was used to estimate the hardness evolution of W–K before and after thermal load tests.

2.3. Characterization

Electron backscatter diffraction (EBSD) with a probe current of 10 nA and an acceleration voltage of 20 kV was applied to acquire the microstructure information on the RD–TD surface of W–K, which was handled via Channel 5 software. The dots with confidence exponent $<0.1$ were removed to perform post-processing, so that the grain size distribution diagram could be acquired. Low-angle grain boundary (LAGB) and high-angle grain boundary (HAGB) are defined as the grain boundary misorientations of $2^-15^\circ$ and over $15^\circ$, respectively.
A scanning electron microscope (SEM) was used to characterize the surface morphology before and after thermal load tests.

3. Results

3.1. Microstructure of the rolled W–K alloy

Figure 3 presents the microstructure information of rolled W–K alloy including grain orientation distribution, grain boundary misorientation distribution and mean grain dimension, which are acquired and processed via EBSD technology and Channel 5 software. The inverse pole figure (IPF) on the RD–TD surface of W–K is colored red, green and blue, which represent the surface grain orientations of (001), (101) and (111) perpendicular to the normal direction and dominated by (111) orientation, see figure 3(a). As shown in figure 3(b), the 289 grains are counted by Channel 5 and the results show that the grain size of rolled W–K is primarily distributed in the range of 0–12 μm and the mean value is about 2.81 μm. The HAGBs and LAGBs, represented by green and blue lines, respectively, (figure 3(c)), are used to distinguish the type of grain boundaries. As shown in figure 3(d), the grain boundary misorientation distribution map distinctly indicates that the grain boundaries of rolled W–K are mainly LAGBs.

To further reveal characteristics of K bubbles, figure 4(a) displays the fracture morphology of rolled W–K alloy. More than 500 bubbles were counted randomly in ten fracture pictures and the size distribution is summarized in figure 4(b). An interesting phenomenon is that the distribution of K bubbles with a mean size of ∼68.5 nm is not random but presents a specific string arrangement along a certain direction, called K bubble string. For more detailed characteristics of K bubbles, please refer to our previous work [34]. In general, K-tube is extremely difficult to observe because it is in a critical state during the processing of W–K plate. In this work, we observed the presence of K-tube in rolled K-W alloy as marked by the yellow dashed outline (figure 4(a)), which clearly demonstrates the formation process of K bubble string. This unique K bubble string structure plays an extremely important role in improving the high-temperature stability and thermal shock properties of W materials, which will be discussed in detail in the following sections. In addition, the typical second phases are analyzed via EDS and the results indicate that the major components are W and K elements, as shown in figure 4(c).
3.2. Thermal conductivity of the rolled W–K alloy

Thermal conductivity is deemed to be one of the key factors affecting the efficiency of heat transfer, which can determine the surface temperature of a sample during thermal load and further affect the thermal load resistance of a material. Consequently, enhancing the mechanical behavior of the material without decreasing its thermal conductivity is extremely important to raise the thermal load resistance of the material. The relationship between thermal conductivity of W–K and test temperature is illustrated in figure 5, where the data of W–0.5TiC [38], W–0.5ZrC [39] and ITER-W [38] are also supplied for comparison. It is found that the thermal conductivity of W–K and ITER-W is almost the same with only minor variations of below 1%. This indicates that the addition of K has little effect on the heat transfer efficiency of PW. However, the addition of TiC and ZrC particles significantly reduces the thermal conductivity, which will reduce the thermal shock resistance of PW. Previous studies have also proved that the existence of ceramic phase in W matrix significantly reduces its thermal conductivity [40–44]. It is widely accepted that the grain size, second-phase particles, grain boundaries, intrinsic defects, etc, are critical factors determining the thermal conductivity of a material. However, Fukuda et al [28] further confirmed that the grain size, boundaries, including both grain and sub-grain boundaries, and defects such as dislocations do not have a significant effect on the thermal diffusivity by comparing the thermal diffusivity of single-crystalline and polycrystalline pure W. Hence, the decrease in thermal conductivity in W–0.5TiC and W–0.5ZrC is possibly because adding the ceramic phases of TiC and ZrC in W can introduce more W/TiC and W/ZrC phase to prevent the heat transfer compared with ITER-W, thereby resulting in a decrease in thermal diffusivity. It should be emphasized that the thermal conductivity of TiC and ZrC as ceramic phases is much lower than that of ITER-W. In addition, the small size (∼68.5 nm) and extremely low content (∼92 ppm [34]) of K bubbles are also considered to be one of the reasons for the smaller impact of K doping on the thermal conductivity of PW.
Thermal shock fatigue resistance of the rolled W–K alloy

Thermal shock tests were performed to investigate the response of W–K to transient thermal shock, and the APD is set to 0.33, 0.44, 0.55 and 0.66 GW m\(^{-2}\), respectively. Figure 6 displays the low- and high-magnification SEM images of the loaded surface of W–K after transient thermal shocks at RT for 100 shots at different APDs with a single-pulse duration of 1 ms. It can be seen from figures 6(a) and (a1) that no surface micro-cracks and obvious coarsening are detected, indicating that the W–K does not produce any surface damage under the thermal shock condition of 0.33 GW m\(^{-2}\). As the APD increases to 0.44 GW m\(^{-2}\), a slight surface roughness and some strip-like structures are observed in the low-magnification SEM image, see figure 6(b). However, no cracks are found after magnification (figure 6(b1)), indicating the cracking threshold of W–K is higher than 0.44 GW m\(^{-2}\). With the
Figure 7. Low- (left) and high-magnification (right) SEM images of the loaded surface of W–K after transient thermal shocks at RT for 100 shots at 0.55 GW m\(^{-2}\) with a single-pulse duration of 1 ms. (a) and (a1) WK-1, (b) and (b1) WK-2, (c) and (c1) WK-3.

Table 1. Comparison of crack threshold between the rolled W–K in this work and other W-based materials in previous reports for 100 pulse thermal loads tests at RT.

| Materials                  | Pulse duration, ms | Crack thresholds (GW m\(^{-2}\)) | Reference |
|----------------------------|--------------------|-----------------------------------|-----------|
| Rolled-WK                  | 1                  | 0.44–0.55                         | This work |
| Forged commercial W–K      | 1                  | 0.19–0.38                         | [7]       |
| Forged + rolled W–K        | 5                  | >0.44                             | [32]      |
| Sintered SPS-WK            | 5                  | 0.37–0.50                         | [30]      |
| Sintered SPS-PW            | 5                  | <0.37                             | [30]      |
| ITER-W                     | 1                  | >0.38                             | [47]      |
| IGP-W                      | 1                  | 0.22–0.33                         | [48]      |
| Sintered SPS-W–0.2ZrC      | 1                  | 0.22–0.33                         | [49]      |
| Rolled W–0.5ZrC            | 1                  | 0.22–0.33                         | [39]      |
| Rolled W–1.0TaC            | 1                  | 0.33–0.44                         | [50]      |
| Rolled SPS-W–Y\(_2\)O\(_3\) | 1                  | <0.60                             | [51]      |
| Forged W–Y\(_2\)O\(_3\)   | 1                  | 0.22–0.33                         | [52]      |
| W–1Ta                      | 1                  | 0.30–0.60                         | [7]       |
Figure 8. Microstructures on the cross-sectional surfaces of W–K. (a) 0.44, (b) 0.55, (c) 0.66 GW m$^{-2}$.

Figure 9. IAMA maps of W–K after thermal shock experiments with an APD of (a) 0.44, (b) 0.55, (c) 0.66 GW m$^{-2}$, and the percentage of recrystallized, substructured and deformed areas in the three samples calculated via Channel 5 software are presented in (d).

APD further increasing to 0.55 and 0.66 GW m$^{-2}$, cracks were visible in both cases and the surface damage becomes more serious as the APD increases (figures 6(c)–(d1)). This suggests that the cracking threshold of W–K is in the range of 0.44–0.55 GW m$^{-2}$. In addition, it is found from the cracking patterns of W–K that only major cracks with low density surround the loaded surface. Generally, two kinds of cracks including major and micro-cracks can be found on the loaded surface of the hot-rolled pure W [45, 46]. The major cracks grow across the loading surface and produce a network and the micro-cracks appear between the major cracks. The generation of major cracks is considered to be related to the brittleness of W below the DBTT, while micro-cracks are mainly intergranular cracks caused by thermal fatigue [46]. Therefore, the formation of major cracks in W–K is caused by brittleness at RT rather than thermal fatigue. It is necessary to emphasize the fact that four samples in total were tested at 0.55 GW m$^{-2}$ and only one sample was cracked. Figure 7 presents the other three samples without cracks at 0.55 GW m$^{-2}$ and are called WK-1, WK-2 and WK-3.
respectively. In order to more accurately evaluate the thermal shock resistance of W–K alloy, table 1 lists the cracking threshold of W–K and other typical W-based materials reported in the literature, wherein the experimental parameters are the same as this work. These data distinctly demonstrate that the rolled W–K alloy has a better thermal load resistance compared to the most advanced W-based materials.

In order to analyze the crack patterns in material depth, cross-sectional morphology analysis was conducted, as shown in figure 8. No cracks are observed on the cross-section at 0.44 GW m$^{-2}$ (figure 8(a)), which is consistent with the results observed on the RD–TD surface. In the case of 0.55 and 0.66 GW m$^{-2}$, transgranular cracking parallel to the heat flux is observed with depths of $\sim 223$ and $\sim 237 \, \mu m$, respectively. Based on previous work, it is known that the grains of rolled W–K are elongated along the RD in the cross-section [34]. Therefore, it can be determined that the cracks parallel to the heat flux are transgranular cracks. Generally, the crack propagation and patterns are also intensely affected by the grain orientation/shape [53]. In addition to crack formation parallel to the heat flux, the rolled W materials show crack formation perpendicular to the heat flux, which can be found in previous reports [39, 50, 53]. The cracks perpendicular to the heat flux usually appear at the depth of transition between compressive and tensile thermal stresses, which depends on the used pulse length and APD, i.e. produced temperature gradient. This is also a direct result of material anisotropy, showing that the cracking resistance along the elongation direction of grains is reduced. These cracks perpendicular to the heat flux act as a thermal barrier, inducing overheating and melting due to the significant decrease in thermal conductivity, which can significantly reduce the lifetime of PFMs. However, only intragranular cracking parallel to the heat flux is observed in the cross-sectional image (figures 8(b) and (c)), indicating obvious transgranular cleavage and stronger cohesion of grain boundaries in the W–K. This is exactly the same crack pattern as our previous swaged plus rolled W–K [32].

It is known that high stress and temperature can occur simultaneously on the sample surface due to repeated (100 cycles) high thermal loads. The extremely high temperature could induce recrystallization and grain growth, thus affecting the thermal fatigue resistance of PFMs. Therefore, the EBSD technology was used to investigate the recrystallization behavior of W–K after thermal load experiments with different APDs. As presented in figure 9, the internal average misorientation angle (IAMA) maps of W–K after thermal load experiments were obtained via Channel 5 software. According to the calculation of IAMA, the identified grains are commonly divided into three states: recrystallized, substructured and deformed. In figures 9(a)–(c), the grains with IAMA below 1$^\circ$ and between 1$^\circ$–7.5$^\circ$ are considered to be recrystallized and deformed states, which are colored blue and red, respectively. The yellow grains are classified as being in a substructured state since their IAMA is under 1$^\circ$ but the misorientation angle between sub-grain and sub-grain is 1$^\circ$–7.5$^\circ$. Substructure is a kind of mosaic structure, which generally refers to the dislocation arrangement and distribution within the crystal. Specifically, the crystal is divided into sub-grains with a small orientation difference, and sub-grain boundaries can be attributed to the ranks or networks of dislocations. That is, the dislocations in the crystal can form ranks or networks by climbing during the high-temperature treatment of rolled W materials, resulting in the appearance of sub-grains. Figure 9(d) summarizes the percentage of recrystallized, substructured and deformed areas of the investigated samples. As shown in figure 9(a), in the case of 0.44 GW m$^{-2}$, the grains show deformed and substructured characteristics and the percentages are 35.5% and 61.9%, respectively. As the APD rises to 0.55 GW m$^{-2}$, the IAMA maps are still dominated by substructured regions and a percentage of up to 89.8%, see figure 9(b). Moreover, a few slightly deformed and recrystallized areas are also found but the percentages are both less than 10%. This suggests that only extremely restricted recrystallization occurs in the thermal shock tests with an APD of 0.55 GW m$^{-2}$. With the APD increasing to 0.66 GW m$^{-2}$ (figure 9(c)), although it is still dominated by substructured regions, the recrystallized percentage has risen to 25.9%. Based on the EBSD results, it can be concluded that recrystallization has occurred in local areas during the high thermal load tests with an APD of 0.33–0.66 GW m$^{-2}$. According to equation (2) in section 2.2, the maximum surface temperature of W–K after a single pulse for 1 ms at 0.66 GW m$^{-2}$ is calculated to be around 1263 $^\circ$C, which is significantly lower than the recrystallization temperature measured in the previous work [34]. However, recrystallization occurred at 0.33 GW m$^{-2}$, corresponding to the maximum surface temperature of 633 $^\circ$C for W–0.5ZrC with excellent mechanical properties [39]. This may be due to the repeated double acceleration of high temperature and stress [39]. Hence, it can be concluded that the W–K alloy has excellent high-temperature stability. This can be attributed to the fact that it is extremely difficult for K bubbles to cause Ostwald ripening and the unique K...
bubble string structure, which will be described in detail in the following sections.

In addition, the recrystallization behavior can also be affirmed via the evolution of hardness because the Vickers micro-hardness is closely related to microstructure of materials. Therefore, the recrystallization process of W–K during thermal shock can also be evaluated through the evolution of hardness. Figure 10 shows the Vickers micro-hardness of W–K before and after thermal load experiments with a different APD. Obviously, the micro-hardness of 430.8–443.2 HV0.2 at 0.33–0.66 GW m⁻² is somewhat lower than 449.3 HV0.2 of the stress-relieved state, but significantly higher than 402 ± 5 HV0.2 of the W–K alloys in the recrystallization state [32]. The decline in hardness at 0.33–0.66 GW m⁻² may be due to the recovery process caused by high temperature, which can remove line and point defects in W–K. Similarly, the rolled PW and W–TaC also have almost the same micro-hardness change trend during the recrystallization process. The micro-hardness of PW and W–TaC drops from 434 ± 2 and 485 ± 2 in the stress-relieved state to 425 ± 3 and 475 ± 2 in the recovery phase, respectively, and then to 351 ± 2 and 410 ± 2 HV0.2 in the recrystallized state [50, 54]. Therefore, it can be concluded that no recrystallization occurred on the surface HV0.2 in the recrystallized state [50, 54]. Therefore, it can be concluded that no recrystallization occurred on the surface.

4. Discussion

Generally, the high thermal load resistance of materials is closely related to their microstructure, mechanical and thermal properties, etc [53]. It can be seen from section 3.2 that the thermal conductivity of rolled W–K is comparable to that of ITER-W and significantly higher than that of the ceramic thermal conductivity of rolled W–K is comparable to that of ITER-W and significantly higher than that of the ceramic. Therefore, the effect of K bubbles with unique string structure on the high-temperature stability and mechanical properties of W materials needs to be discussed in detail. Before that, we should first clarify the formation process and characteristics of K bubble strings.

4.1. Formation process and characteristics of K bubble strings

In the preparation of W–K, the tungsten trioxide powder was doped into a mixed solution of potassium silicate and aluminum nitrate. At this stage, W lattice mainly exists in the form of β-W with an atomic radius (r) of 0.218 nm. Therefore, aluminum (Al), silicon (Si) and oxygen (O) atoms with r of 0.142, 0.117 and 0.074 nm, respectively, can enter and leave the β-W lattice freely, while K atoms with r of 0.235 nm cannot enter the β-W lattice. The r is calculated by equation (3):

\[ r = \frac{\sqrt{3}}{4} a, \]

where a is the lattice constant of an atom. K atoms can only be brought into the W lattice via the transition from β-W to α-W during subsequent hydrogen reductions. The high mobility of W atoms during the transition from β-W to α-W can lead to K atoms being trapped in the α-W lattice, and it is difficult for those K atoms entering into the α-W lattice to escape because the r of K atom is higher than that of W atom. Therefore, most of Al, Si and O are volatilized but a certain amount of K in the form of ellipsoid K-pores can be retained in the subsequent sintering process. It needs to be stated that α-W is what we usually refer to as metal W, and β-W is a sub-stable phase structure of the metal W, which is the product of the reduction of WO₃ by hydrogen and the reaction is as follows [55]:

\[ 380^\circ C, \text{WO}_3 + 0.1\text{H}_2 \rightarrow \text{WO}_{2.90} + 0.1\text{H}, \]

\[ > 440^\circ C, \text{WO}_{2.90} + 2.9\text{H}_2 \rightarrow \beta_{-}\text{W} \cdot +2.9\text{H}_2\text{O}, \]

\[ > 630^\circ C, \beta_{-}\text{W} \rightarrow \alpha_{-}\text{W}. \]

Based on the Rayleigh model, a schematic diagram of the formation process of K bubbles is shown in figure 11. First, the ellipsoidal K-pores will be transformed into K-tubes with a certain aspect ratio (l/d) during hot rolling. Then, the surface instability of wavelength λ is able to be formed along the direction of the K-tube at elevated temperature. When l/d is greater than 2π, the K-tubes will break up into K bubble strings due to their aggravated instability. Once K bubbles are formed during hot rolling, they will grow to a balance dimension in the subsequent high-temperature annealing. The dimension is decided via the equilibrium between the Laplace pressure generated via surface tension of W and the pressure in bubbles produced via K vapor. According to Laplace equation (7),

\[ \Delta P = \frac{2\gamma}{R}, \]

wherein \( \Delta P \) is the pressure difference between two sides of the interface; \( \gamma \) and \( R \) are the interface tension and curvature radius, respectively. K bubbles are in equilibrium when the pressure in K bubbles and Laplace pressure are equal. In the case of ideal gas behavior of K and globular bubbles, equation (7) can be converted to (8):

\[ \frac{3nRT}{4\pi r^3} = \frac{2\gamma}{r}. \]

Further detailed characteristics of K bubbles can be found in our previous work [34].

4.2. K bubbles induce excellent high-temperature stability

It is widely accepted that the existence of the doped second phase can effectively improve the high-temperature stability of materials by inhibiting the movement of grain boundaries and dislocations. Based on the linear elastic theory, interaction energy (E) between the second phase and dislocations can be expressed by equation (10):

\[ E = \frac{3\pi nRT}{8\pi \gamma}. \]
in which $G$ and $r_0$ are the shear modulus of the material and the radius of dislocation central region, respectively. $b$ and $a$ stand for the Burgers vector and a spacing between the second phase and dislocations, separately. When the dislocations move near the second phase, the second phase will become highly attracted to the dislocations. Hence, doping carbides, oxides and K bubbles can effectively improve the high-temperature stability of PW by pinning grain boundaries and dislocations. However, it is necessary to state that the K bubbles as gaseous second phases have a better effect on improving the high-temperature stability of PW than the solid second phases. The reasons can be summarized with the following two points: (i) it is difficult for Ostwald ripening of K bubbles to occur at high temperature; (ii) unique K bubble string structure.

For (i), it is well known that the size of second phase particles plays a very important role in the final dispersion strengthening effect [56]. That is, if the dispersed second phases grow due to Ostwald ripening, the disconnected interface between the second phases and grains will turn into the preferred locus for crack formation and thus reduce the strengthening effect. However, it is easy for traditional solid second phases to initiate Ostwald ripening at high temperature. This is primarily due to the diffusion and dissolution of tiny solid particles on large particles, which is achieved by diffusion of solid second phase atomic lattice [57]. However, for K bubbles, it is extremely difficult for the Ostwald ripening to occur at high temperature because they hardly dissolve in W and their lattice diffusion in W is also extremely difficult [57]. A previous study has also confirmed this conclusion [58]. For example, W–K wires were annealed at 1810 °C–2640 °C by Liu et al and it was found that the distribution and size of K bubbles hardly changed [58].

For (ii), although the dispersed second phase particles can effectively improve the high-temperature stability of PW by pinning the grain boundaries and dislocations, they cannot control the preferred orientation during grain growth at high temperature. This limits the strengthening effect of second phases, and thus leads to the formation of equiaxed grains in conventional W-based material after recrystallization. For example, Xie et al [39] investigated the recrystallization behavior of rolled W–0.5ZrC and the results indicate that the elongated grains with an aspect ratio of around 4 in the stress-relieved state become equiaxed grains with an aspect ratio of around 1 after annealing at 1600 °C for 1 h. However, K bubble strings distributed along RD can not only pin the grain boundaries and dislocations, but also control the growth direction of grains at high temperature. The K bubble strings parallel to each other can effectively inhibit the growth of grains perpendicular to the RD, which will lead to a further increase in the aspect ratio because the growth constraint of grains along the RD is smaller. As a result, these grains will be connected to each other in the form of a dovetail splicing structure after high-temperature annealing. This view is confirmed by our previous work [34]. The rolled W–K was annealed at 1600 °C and 1800 °C and the results show that the elongated grains with an aspect ratio of around 4 in the stress-relieved state developed into elongated grains with an aspect ratio of around 7 and 5 after annealing at 1600 °C and 1800 °C, respectively [34]. It is worth mentioning that the recrystallization percentage of W–K is still less than 10% after 1800 °C annealing [34]. Although the grain size of W–K will also increase after high-temperature annealing, the distinctive dovetail splicing structure with a certain aspect ratio can better improve the high-temperature stability of the material compared with the equiaxed grain structure. Therefore, K bubbles as gaseous second phases may have a better effect on improving the high-temperature stability of PW compared with the traditional solid second phase.

4.3. K bubbles induce superior mechanical properties

Generally, the appearance of cracks during thermal fatigue tests is primarily due to the thermal tensile stresses caused via temperature gradient [59]. The cracks will be formed when the thermal tensile stress produced in the cooling stage is
greater than the ultimate tensile strength (UTS) of materials. Therefore, the addition of second phases can effectively improve the thermal shock resistance by improving the UTS of materials.

At RT, K bubbles have a similar strengthening mechanism with the traditional solid second phases, i.e. fine grain strengthening and second phase (K bubble) strengthening. In the case of fine grain strengthening, the yield strength ($\sigma_{FG}$) can be calculated by the Hall–Petch equation (11):

$$\sigma_{FG} = \sigma_0 + \frac{k}{\sqrt{d}}, \quad (11)$$

where $k$ and $d$ are the Hall–Petch constant and grain size, respectively. For $d$, it should be emphasized that the surface of counted grains should be perpendicular to the tensile loading direction. For example, if the tensile loading direction is congruent with the RD, then the grain size should be counted based on the TD–ND surface; $\sigma_0$ is lattice friction stress of dislocations. Therefore, the yield strength increment ($\Delta \sigma_{FG}$) of W–K compared to PW arising from fine grain strengthening can be obtained via equation (12):

$$\Delta \sigma_{FG} = k\left(\frac{1}{\sqrt{d_{WK}}} - \frac{1}{\sqrt{d_{PW}}}\right). \quad (12)$$

It can be deduced that $\Delta \sigma_{FG} > 0$, considering that the addition of K can refine W grains and thus result in $d_{WK} < d_{PW}$.

With regard to K bubble strengthening, the yield strength increment ($\Delta \sigma_{KB}$) can be determined via a dispersed-obstacle model based on Bacon et al [60], i.e. the BKS model (13):

$$\Delta \sigma_{KB} = MG0b\sqrt{Nd} \quad (13)$$

where $M = 3.06$ in bcc metals [61], is the Taylor factor; $\alpha$ is the barrier strength and the value is a constant 0.3 [62]; $G$ and $b$ are the shear modulus and Burgers vector, 160 GPa [63] and 0.274 in bcc metals [64], respectively. $d$ and $N$ are the mean size of K bubble and number density, separately. Therefore, the strength difference ($\Delta \sigma$) of W–K and PW can be calculated by equation (14):

$$\Delta \sigma = k\left(\frac{1}{\sqrt{d_{WK}}} - \frac{1}{\sqrt{d_{PW}}}\right) + MG0b\sqrt{Nd} \gg 0. \quad (14)$$

At high temperature, the K bubbles have a better strengthening effect than the traditional solid second phases. This is because the Ostwald ripening of K bubbles hardly occurs and K bubbles have a distinctive dovetail splicing structure with a certain aspect ratio. Nano-sized K bubbles still maintain an excellent strengthening effect at high temperature and the distinctive dovetail splicing structure can improve the strength of W–K by inhibiting the propagation of cracks. Therefore, K bubbles are more effective in improving the high-temperature strength of PW compared with the traditional solid second phases.

4.4. K bubbles inhibit the formation and propagation of cracks during thermal shock

According to the material fracture theory, it is known that the ultimate behavior of fatigue failure of metal materials is fracture, which is mainly due to their loss of stability before crack propagation. During the stress cycle, fracture occurs when the maximum stress intensity factor $K_{I_{max}}$ at the front of the crack exceeds the fracture toughness $K_{IC}$ of the material, which can be expressed by equation (15):

$$K \sqrt{a_{IC_{max}} \rho_{max}}, \quad (15)$$

where $Y$ and $\sigma_{max}$ are the crack-shape factor and the maximum value of cyclic stress, respectively; $a$ represents the crack length before fracture. In addition, $\sigma_{max}$ is closely related to the geometry of the crack front, that is,

$$\sigma_{max} \sqrt{\frac{a}{\rho_{max}}}, \quad (16)$$

where $\rho$ stands for the curvature radius of the crack front end, as shown in figure 12. Therefore, the decrease in $a$ and increase in $\rho$ can effectively inhibit the formation and propagation of cracks. It is well known that K bubbles exist in gaseous form at high temperature because its boiling point is 760 °C [65]. Therefore, the vapor pressure in the K bubbles will rise rapidly with the temperature during thermal shock, which can lead to significant compressive stress between K bubbles and W matrix. The crack front end is bound to encounter the stress field generated by the expansion of K bubbles when it propagates along the grain boundaries. This will reduce the driving force that promotes crack propagation, resulting in a tendency for the front end of the crack to close, i.e. the passivation effect. This can effectively increase the curvature radius of the crack front end, i.e. the value of $a/\rho$ is reduced. As a result, the decrease in $K_{I_{max}}$ improves the cracking resistance of W–K during thermal shock.

In addition, the deformability of K bubbles at high temperature is one of the key factors inhibiting crack formation. Tensile and compressive stresses caused by alternating changes in

![Figure 12. Schematic diagram of micro-crack formation induced by second phase during grain boundary slippage.](image-url)
temperature during thermal shock can lead to rapid expansion and contraction of the W grains, which will induce plastic deformation dominated by grain boundary slip. As shown in figure 13(a), the tensile stress generated during the cooling process causes the grain boundaries to slip away from the traditional solid second phase and this separation will form holes, i.e. micro-crack. It is extremely easy for this micro-crack to expand under alternating loads, and thus results in cracking during thermal shock. However, in the case of W–K, this micro-crack source generally cannot appear during thermal shock due to the deformability of K bubbles. As presented in figure 13(b), K bubbles can be deformed with the slippage of grain boundaries to avoid the emergence of micro-crack sources.

K bubble strings also play an important role in inhibiting the propagation of cracks during thermal shock. According to the description in section 4.2, the existence of K bubble strings can cause the W grains with a certain aspect ratio to connect with each other in the form of a dovetail splicing structure rather than equiaxed grains after high-temperature annealing. The cracks can easily extend along the weak grain boundaries to the W matrix when the traditional W-based alloy is recrystallized to form equiaxed grains during thermal shock. However, the propagation of cracks in W–K is more difficult because the grains of the dovetail splicing structure caused by K bubble strings have stronger grain boundary cohesion. This will result in the cracks only consuming more energy to penetrate the grains, rather than propagating along the weak grain boundaries of equiaxed grains like traditional W materials, see figures 8(b) and (c). Therefore, K bubble strings can more effectively restrain the propagation of cracks during thermal shock compared with the traditional solid second phase particles.

In summary, there are four reasons W–K alloy exhibits excellent thermal shock resistance: (i) the doping of K does not reduce the thermal conductivity of PW; (ii) the doping of K can improve the high-temperature stability of PW, especially the distinctive dovetail splicing structure with a certain aspect ratio after high-temperature annealing; (iii) the doping of K can evidently enhance the mechanical properties of PW; (iv) K bubbles can effectively inhibit the formation and propagation of cracks during thermal shock.

5. Conclusion

In this work, a newly developed large-volume W–K plate with a thickness of 15 mm and a weight of 25 kg by powder metallurgy plus hot rolling was prepared in order to meet the requirements of ITER in engineering application. To shed light on the effect of K bubbles on thermal shock performance, the transient-state thermal shock resistance of W–K with a single-pulse duration of 1 ms for 100 shots at RT was appraised. The absorbed power density is set to 0.33, 0.44, 0.55 and 0.66 GW m$^{-2}$, respectively. In addition, the thermal conductivity, microstructure and micro-hardness before and after thermal shocks are characterized. The major conclusions are summarized below:

(a) The cracking threshold of rolled W–K is 0.44–0.55 GW m$^{-2}$, suggesting that W–K alloy has better transient thermal shock resistance compared with the most of advanced W-based materials.

(b) The doping of K does not decrease the thermal conductivity of PW, which is possibly due to the small size (∼68.5 nm) and extremely low content (∼92 ppm) of K bubbles.

(c) No sign of recrystallization is evidently observed on the surface of the W–K samples during thermal shock at 0.33–0.66 GW m$^{-2}$ because the K bubble strings can greatly improve the high-temperature stability of W material.

(d) The doping of K can significantly enhance the mechanical performance of W material by fine grain strengthening and K bubble strengthening mechanism.

(e) Deformable gaseous K bubbles can effectively restrain the formation and propagation of cracks during thermal shock.

Therefore, it can be concluded that the rolled-WK alloy is a highly promising candidate for PFMs. These studies are beneficial for building a trusted ITER database for the advanced W-based materials, which is able to provide a useful reference for numerical simulation under high heat flux and the selection of future PFMs.

Acknowledgments

This work was supported by the National Natural Science Foundation of China (Grant Nos. 11905137 and 51801171), the National MCF Energy R&D Program (Grant No. 2019YFE03130002), the ITER National Magnetic Confinement Fusion Program (Grant No. 2014GB123000), the Fundamental Research Funds for the Central Universities (Grant No. 2021MJ051) and the Interdisciplinary Innovation Program of North China Electric Power University (Grant No. XM2112355).

CRediT authorship contribution statement

Xiaolei Ma: conceptualization, methodology, investigation, writing-original draft. Fan Feng: review, thermal shock.
tests. Xiaoxin Zhang: review and editing, supervision, funding acquisition. Ting Wang: review and microstructure characterization. Xiang Liu: review, thermal shock tests. Wei Lv: review, funding acquisition. Shouting Lang: review, Vickers micro-hardness tests. Changchun Ge: conceptualization and review. Qingzhi Yan: conceptualization, methodology, review and editing.

Data availability

The data that support the findings of this study are available upon reasonable request from the authors.

Declaration of interest statement

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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