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Thermal shock behavior of W–ZrC/Sc_2O_3 composites under two different transient events by electron and laser irradiation

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ABSTRACT

The transient thermal shock behaviors of W–ZrC/Sc_2O_3 composites with different ZrC contents were evaluated using transient thermal shock test by electron and laser beams. The effects of different ZrC doping contents on the surface morphology and thermal shock resistance of W–ZrC/Sc_2O_3 composites were then investigated. Similarity and difference between effects of electron and laser beam transient heat loading were also discussed in this study. Repeated heat loading resulted in thermal fatigue of the irradiated W–ZrC/Sc_2O_3 samples by thermal stress, leading to the rough surface morphologies with cracks. After different transient thermal tests, significant surface roughening, cracks, surface melting, and droplet ejection occurred. W–2vol.%Sc_2O_3 sample has superior thermal properties and greater resistance to surface modifications under transient thermal shock, and with the increasing ZrC content in W alloys, thermal shock resistance of W–Zr/Sc_2O_3 sample tends to be unsatisfied.

1. Introduction

Plasma facing materials (PFMs) require the ability to withstand extreme temperature, irradiation, and thermal stress in fusion reactors (ITER and future DEMO reactors). Tungsten (W) is currently considered as one of the most promising candidates for PFMs due to its high melting point, high strength at high temperatures, good thermal conductivity, low thermal expansion coefficient, high sputtering threshold energy and limited activation under neutron irradiation [1–4]. However, tungsten undergoes serious embrittlement at low temperature during recrystallization and during irradiation [5–8]. Moreover, the embrittlement significantly decreases the thresholds of the crack initiation and enhances crack propagation, thus limiting the application of tungsten in the future fusion devices and reactors. In addition to steady-state heat loading of 5–20 MW/m^2, PFMs are exposed to transient high heat loads, such as edge localized modes (ELMs, in the order of 1 MJ/m^2 for ~0.5 ms) and disruptions (several 10 MJ/m^2 for several ms) during plasma operation in fusion device, which causes serious material degradation [9–11]. The lifetime of materials and components is primarily limited by these transient high heat loads [12]. High temperature gradient and high thermal stresses developed during transient events can lead to material recrystallization, grain growth, cracking, surface melting, evaporation, droplet ejection, and fatigue fracture, leading to fatal damages of PFMs [13,14]. Therefore, the behavior and thermal shock resistance of tungsten and its alloys under transient high thermal loads is an important aspect for material analysis.

To investigate the thermal shock behavior of W and W alloys, different types of heat sources, such as plasma gun, electron beam, and pulse laser facilities are performed separately or simultaneously to stimulate ITER-relevant high heat-fluxes, such as ELM effects on PFMs. M. Wirtz et al. [15] combined high-flux hydrogen-plasma (Pilot-PSI) and subsequently transient electron beam heat

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load (JUDITH 1) in their study and later found that preloading with high-flux hydrogen-plasma has significant influence on the thermal shock behavior of W in terms of crack distance, width, and depth as well as cracked area. Similarly, G. De Temmerman et al. [16] also investigated the effect of high-flux H/He plasma exposure on W damage due to transient heat loads using high-flux plasma (Pilot-PSI) and a high-power laser (LASAG FLS 352-302). Moreover, Bo Huang et al. [12] and Z.M. Xie et al. [14] also investigated the effects of different dispersed second-phase particles to W alloys with the electron beam test facility on the thermal shock characterization and behavior as a judge standard on thermal shock resistance because some dispered second-phase particles can effectively solve the embrittlement problems of W alloys (such as La_2O_3, Y_2O_3, TiC, and ZrC) [17–20] through effective inhibition of recrystallization and improvement of the high-temperature strength and creep resistance by hindering grain boundary (GB) sliding and stabilizing the micro-structure when exposed to high temperatures [21,22] M.Y. Zhao et al. [11].

Based on our previous study that adding Sc_2O_3 particles dopant could not only refine the grains and increase the W alloy density but could also improve the strength of the samples [23], we also added some ZrC particles and attempted to improve the toughness and ductility of W alloys as well as the stability resistance of the alloy against high-energy particle and ion irradiation. In this study, thermal shock tests of W–ZrC/Sc_2O_3 composites under two different transient events by electron and laser irradiations were performed. Combining with the microstructure, thermal conductivity, relative density, and thermal shock behavior, effects of different ZrC doping contents on the thermal shock response were presented and discussed.

2. Experimental

Different ZrC contents of W–ZrC/Sc_2O_3 composites (i.e., W–2vol.%Sc_2O_3, W–1vol.%ZrC/2 vol%Sc_2O_3, W–3vol.%ZrC/2 vol% Sc_2O_3, and W–5vol.%ZrC/2 vol%Sc_2O_3) were produced through powder metallurgy (PM) and subsequent spark plasma sintering (SPS) at 1700 °C and 14.3 kN, which can produce fine-grain and high-density materials at a relatively low temperature [24,25]. Afterward, the microstructural characteristics and properties of the alloy were characterized. Thermal conductivity test was performed using a laser flash thermal analyzer (LFA 457, Germany). Then, thermal conductivity (λ) was calculated from the thermal diffusivity (α), specific heat (C_P), and density (ρ) through the expression, $\lambda = \alpha C p \rho$. Specific heat capacity was determined with the theoretical rule of mixtures according to the following formula:

$$C_P = \frac{V_W \times \rho_W \times C_{PW} + V_{ZrC} \times \rho_{ZrC} \times C_{PZrC} + V_{Sc2O3} \times \rho_{Sc2O3} \times C_{PSc2O3}}{\rho}$$

V_W, V_{ZrC}, and V_{Sc2O3} are the volume fraction of W, ZrC, and Sc_2O_3, respectively, whereas C_{PW}, C_{PZrC}, and C_{PSc2O3} are the specific heat values of W, ZrC, and Sc_2O_3, respectively. The data of C_{PW}, C_{PZrC}, and C_{PSc2O3} at different temperatures were obtained from Ref. [26].

SPS-sintered samples were machined to 10 mm × 10 mm × 1 mm via wire-electrode cutting. Afterward, the polished W–ZrC/Sc_2O_3 samples were subjected to electron beam irradiation as heat loading experiments by an electron beam irradiation test simulator of Research Institute for Applied Mechanics of Kyushu University [9,27]. The experiment was conducted with the following irradiation condition: repeated 2 s irradiation and 7.5 s rest in one cycle of 9.5 s for 100 pulses in vacuum. Samples were mechanically fixed on copper block actively cooled with water. During the experiment, the temperature of cooling water was maintained at 40 °C. Electron beam is controlled by changing the bias voltage of the Wehnelt, which made it possible to rapidly start up and shut off the electron beam [28]. In this case, the energy of the electron beam was 20 keV and the beam diameter was 2.5–3 mm. Heat flux was evaluated as an average value at an irradiated area of approximately 40 MW/m^2 by the beam diameter and net electric current of the electron beam irradiated. And the net current was measured by applying a bias voltage to the sample to suppress the secondary electron induced by the electron irradiation [29]. The surface temperature of the irradiated region was measured by two-color optical pyrometers (400 °C–1000 °C, 1000 °C–3100 °C), approximately ranging from 600 °C to 1300 °C.

Before the short transient events by laser irradiation, samples cut into cubes (4 mm × 4 mm × 2 mm) were also polished to a mirror finish. Afterward, transient high-heat loading tests were performed in a test facility LSW-1000 at room temperature (RT) using laser beam with the power frequency of 15 Hz. Ar gas was used as the shield gas and the cooling medium. The laser beam was scanned across the surface in a straight line at 100 mm/min as a single pulse. The diameter of the laser spots was 1.5 mm, and the irradiation time was 2 ms. The energy of the laser beam is Gaussian profile [10] and the energy in the middle of the laser beam was the highest. Laser power density was evaluated by the electric current, irradiation time, and the power frequency through a special formula. Different electric currents reading of 60, 90, and 120 A had the corresponding laser power density of 50, 75, and 100 MW/m^2, respectively.

Laser Particle Size Analyzer (MS–2000, England) was performed to analyze the particle size distribution of original powders and as–prepared powder. The relative density of the sintered samples was measured using Archimedes method. Meanwhile, Vickers microhardness was obtained with MH–3L by measuring from the center to the sample edges with a loading weight of 300 g held for 15 s. The fractured surface was obtained by breaking up the samples at RT artificially, and field emission scanning electron microscope (FE–SEM; SU8020, Japan) was employed to observe the fracture morphology. Transmission electron microscopy (TEM; JEM–2100F, Japan) was also performed to observe the microstructure of the SPS-sintered W–3vol.%ZrC/2 vol%Sc_2O_3 composite prepared with ion-thinning technology because of its high second phase content which was easier to be observed. After the heat load tests, changes in the surface morphology of W–ZrC/Sc_2O_3 composites were observed by FE–SEM, and energy dispersive X-ray spectroscopy (EDS) was employed to characterize the microstructures of irradiated surface.

3. Results and discussion

3.1. Characterization of powders

Before sintering, W, ZrC, Sc_2O_3, and the as–prepared powder (W–3vol.%ZrC/2 vol%Sc_2O_3) were measured through LPSA. The medium particle diameter of W, ZrC, Sc_2O_3, and as–prepared powders were 3.954 μm, 3.788 μm, 5.064 μm, and 2.228 μm, respectively, as shown in Fig. 1a. The width of particle size distribution was also analyzed by formula: (D90-D10)/D50, with values were 6.763, 5.831, 2.800, and 8.575, respectively. The larger the value was, the wider the distribution interval of powders was. After the ball milling for 40 h, the average particle size of the mixed powder decreased obviously comparing with the medium particle diameter. However, high-energy ball milling also led to some agglomerations, which was proved by the small pink peak of as–prepared powders curve in the right side.

3.2. Characterization of sintered samples

TEM analysis was performed to characterize the dispersion of the ZrC and Sc_2O_3 dispersed phases, as shown in Fig. 1b. The TEM bright field image includes the inset of the selected area electron