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High heat load properties of high purity CVD tungsten

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Abstract

High heat load experiments using electron beams were carried out to investigate the performance of chemical vapor deposition-tungsten coated molybdenum (CVD-W/Mo) under fusion relevant conditions. Microstructural observation showed that no crack had been formed after 300 shot heat load (23 MW/m^2 for 30 s, typical surface temperature: 800 °C). The results indicate that the CVD-W/Mo had good thermal fatigue property under the cyclic heat load. Above 1300 °C at which the recrystalization would start cracks were formed easily by thermal stress. Crack propagation was apt to be less than that of powder metallurgy tungsten in the temperature range. It is considered that the high purity of CVD-W suppressed the embrittlement due to the recrystallization.

1. Introduction

Tungsten is a candidate material for high heat flux components in future fusion devices because of their good thermal properties and low sputtering yield [1]. However tungsten has disadvantages which relate to the brittleness and the heavy weight. One possibility to overcome these disadvantages is coating lighter materials with tungsten that have high melting temperatures such as molybdenum [2,3]. In the present study, heat load experiments on chemical vapor deposition-tungsten coated molybdenum (CVD-W/Mo) were carried out to investigate the performance under cyclic heat loads.

2. Experimental

2.1. Samples

The samples used in this work were high purity CVD-W (purity 99.99998%) on Mo substrate delivered by Tokyo Tungsten Co., Ltd. The dimension of the sample was 10 mm × 10 mm × 6 mm (1 mm CVD-W and 5 mm Mo substrate). Heat treatment for 1 h at 1000 °C in a vacuum was performed to stabilize the microstructure of the samples. Microstructural observation on the cross-section showed that the CVD-W layer has a columnar structure which is perpendicular to the surface and did not have defects at the joint interface. The grain size at the surface, i.e., diameter of a column of the CVD-W was 2–5 μm.

Powder metallurgy tungsten (PM-W) samples (purity 99.95%), with dimensions of 10 mm × 10 mm × 5 mm, were prepared to be compared with CVD-W/Mo. The grain size of PM-W used was about 5 μm.

2.2. Heat load experiments

Heat load experiments have been done with an electron beam irradiation test simulator in the Research Institute for Applied Mechanics (RIAM), Kyushu University. The electron beam was controlled by changing the bias voltage of the Wehnelt, which allows to start up and shut off the electron beam quickly [4]. The electron beam diameter was about 2–2.2 mm. The samples were mechanically fixed on a copper heat sink actively cooled with water. The surface temperature was measured with two-color optical pyrometers (450–1100 °C, 1100–3100 °C) with a diameter of 1 mm on a sample. The experimental condition of the cyclic heat load is

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Table 1
Cyclic heat load experimental conditions

Sample	Heat flux (MW/m^2)	Duration (s)	Desired peak temperature (°C)	Total number of shots
CVD-W/Mo PM-W	23	30	800	300
CVD-W/Mo PM-W	50	30	1300	150
CVD-W/Mo	100	0.1, 1.2, 30	<450, 800, 1800	30, 150, 75

shown in Table 1. These samples were heated from room temperature. Changes of surface morphology after the heat load were observed by scanning electron microscopy (SEM).

3. Results

3.1. Cyclic heat load tests at 23 MW/m^2 for 30 s

The peak surface temperature of CVD-W/Mo and PM-W were between 700 and 800 °C and between 750 and 850 °C, respectively. The peak temperature did not change during the cyclic heat load experiments. Fig. 1 shows the SEM micrographs observed on the surface of the PM-W. After 50 shots, cracks with a width of 2 μm and with a length of roughly 10 μm, were observed on the surface of the PM-W. After 300 shots, in addition to these cracks, transgranular fractures with a width of 0.5 μm and with a length of 15 μm were observed. Fig. 2 shows SEM micrographs observed on the surface of the CVD-W/Mo after the heat load. Visible surface modification were not observed on the CVD-W/Mo after 150 shots. However, exfoliation of parts of grains was observed after 200 shots. After 300 shots, surface morphology showed a similar structure to that after 200 shots.

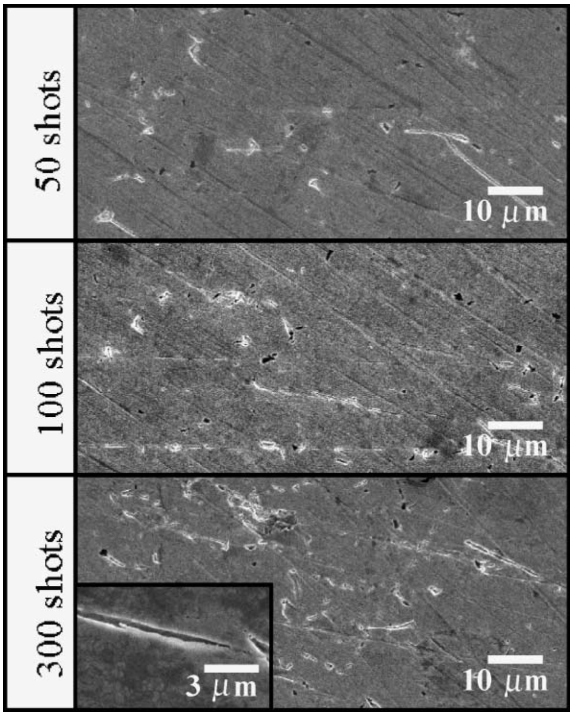

Fig. 1. SEM micrographs of the PM-W loaded at a heat flux of 23 MW/m^2, 30 s (surface temperature: 750–850 °C).

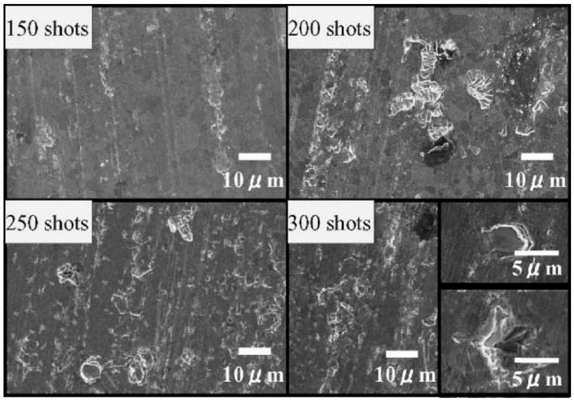

Fig. 2. SEM micrographs of the CVD-W/Mo loaded at a heat flux of 23 MW/m^2, 30 s (surface temperature: 700–800 °C).

3.2. Cyclic heat load tests at 50 MW/m^2 for 30 s

The peak surface temperatures of CVD-W/Mo and PM-W were between 1250 and 1350 °C and between 1280 and 1380 °C, respectively. The peak temperature did not change during the cyclic heat load experiments. Fig. 3 shows the SEM micrographs observed on the surface of the CVD-W/Mo and the PM-W. Cracks were not formed in both samples below 10 shots. After 50 shots, cracks caused by intergranular fracture (width of about 6 μm) were observed in the loaded area in both materials. The size of the cracks on the CVD-W/Mo after 150 shots was still as small as the cracks after 50 shots. On the other hand, a crack with a width of 20 μm and a length of 600 μm was formed in the loaded area on the PM-W after 150 shots.

3.3. Cyclic heat load tests at 100 MW/m^2 for 0.1, 1.2 and 30 s

Cyclic heat load tests with a heat flux of 100 MW/m^2 were performed on CVD-W/Mo. The loading durations for the samples were 0.1, 1.2 and 30 s, respectively. The peak surface temperatures were below 450 °C (outside of the measuring range), between 700 and 850 °C and between 1700 and 1850 °C, respectively. The peak tem-

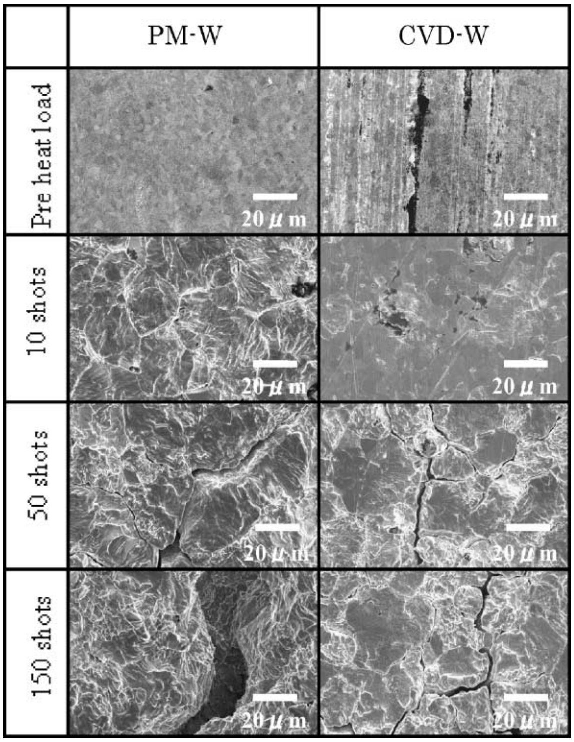

Fig. 3. SEM micrographs of the surface of CVD-W/Mo (surface temperature: 1250–1300 °C) and PM-W (surface temperature: 1280–1380 °C) after heat load tests at a heat flux of 50 MW/m^2, 30 s.

perature did not change during the cyclic heat load experiments. Fig. 4 shows the SEM micrograph observed on the CVD-W/Mo after 30 shots with a beam duration of 0.1 s. As shown in Fig. 4, the net-like cracks were formed in the loaded area between the 21st and 30th shot. As shown in Fig. 5, parts at the surface of the CVD-W exfoliated which was loaded with a beam duration of 1.2 s. Although this exfoliation was enhanced up to 30 shots, loss of surface by exfoliation has not been increased up to 150 shots. Fig. 6 shows the surface of the CVD-W/Mo after 75 shots with a beam duration of 30 s. A crack with a width of 20 μm, and a length of 400 μm, was formed in the surrounding area in between the 51st and 75th shot. At the center of the loaded area, no cracks were observed, but surface modifications due to plastic deformation were detected.

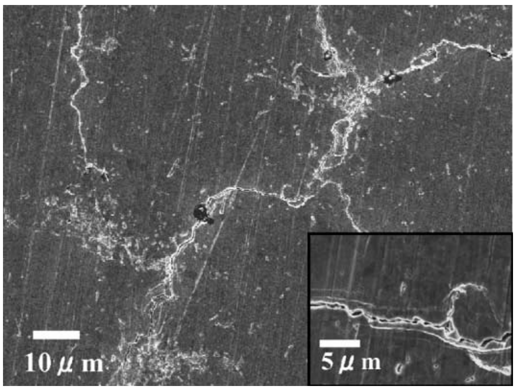

Fig. 4. SEM micrographs of the surface of CVD-W/Mo after 30 shots, 100 MW/m^2, 0.1 s (surface temperature: <450 °C).

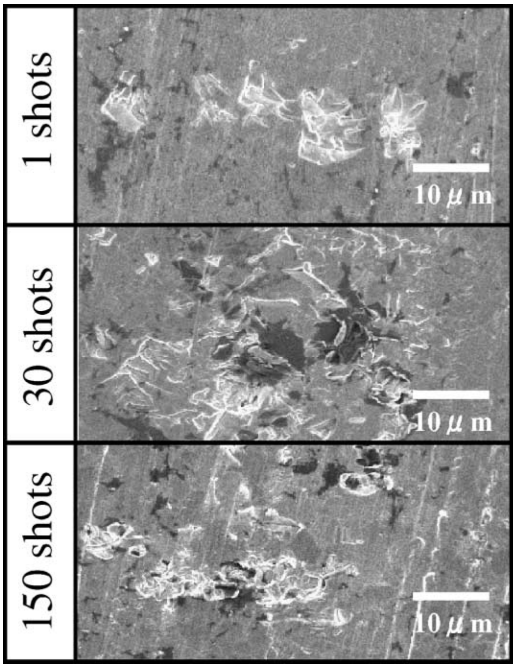

Fig. 5. SEM micrographs of the CVD-W/Mo loaded at a heat flux of 100 MW/m^2, 1.2 s (surface temperature: 700–850 °C).

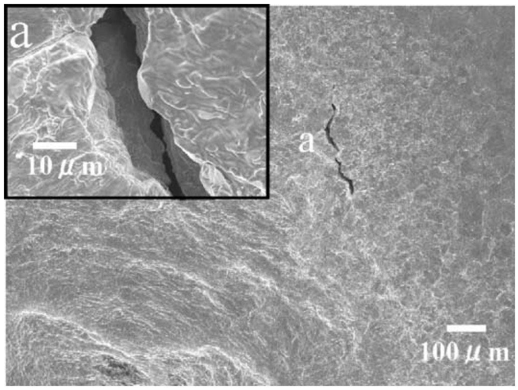

Fig. 6. SEM micrographs of the CVD-W/Mo after 75 shots, 100 MW/m^2, 30 s (surface temperature: 1700–1850 °C). The center of the loaded area is on the left side of the bottom of this figure.

4. Discussion

Exfoliation of parts of grains was observed on the surface of the CVD-W/Mo where the peak temperature by heat loading was above 700 °C. This exfoliation occurred most easily at the loading condition of 100 MW/m^2 in this work. Accordingly, it can be considered that thermal stresses and increasing temperature caused the exfoliation. But loss due to the exfoliation did not increase with increasing heat load. This exfoliation did not occur on the surface of PM-W. The reason for this seems to be that PM-W has rolled structures, which are composed of deformed grains. These grains have complicated boundaries. In this temperature range, it has implications that combinations of the grains of PM-W are superior to that of CVD-W.

The net-like cracks were formed on the CVD-W/Mo surface by the 0.1 s shots between the 21st and 30th pulse at 100 MW/m^2 heat load (the peak temperature was below 450 °C). But the net-like cracks were not formed even after 150 shots at 100 MW/m^2 for 1.2 s (the peak temperature was around 800 °C). In both experiments, the heating rates of these samples were equivalent. Consequently these results indicate that the formation of cracks is independent from heating rates but dependent on the peak temperature. Linke et al. [5,6] reported good thermal shock-resisting properties of the tungsten material when heated up to at a temperature above the DBTT (100–400 °C). The present experimental data agree with their results.

It is well known that embrittlement due to segregation of impurities at the grain boundaries occurs above the recrystallization temperature (around 1300 °C) in tungsten [7]. The cracks due to intergranular fracture were observed on the surface of the samples, where the peak temperature is above the recrystallization temperature. The cracks were observed in the loaded area in both PM-W and CVD-W/Mo, which have been loaded 50 times by 50 MW/m^2 pulses for 30 s. In the cyclic heat-load test at 100 MW/m^2 for 30 s, cracks were observed in the surrounding area of the loaded area on the CVD-W/Mo after 75 shots. The temperature in the surrounding area was below 1800 °C during the heat load. Considering the number of shots until cracks were formed, the location of cracks and the incident heat flux, cracks could be easily formed in the temperature range between 1300 and 1800 °C. This difference was attributed to the changes of mechanical properties of tungsten. Since the yield strength of W at around 1800 °C decreases to 25% of the yield strength at 1300 °C [8], thermal stresses were released by plastic deformation at the loaded area as shown in Fig. 6. The bending strength of PM-W is higher than that of CVD-W from room temperature to around 1000 °C, [9], however, crack propagation in the recrystallized CVD-W was apt to be less than that of the recrystallized PM-W above 1300 °C. These results indicate that the high purity CVD-W is superior to the PM-W in the mechanical properties above the recrystallization temperature.

5. Conclusions

Heat loads experiments have been done to investigate the performance under cyclic heat loads and the load limit of CVD-W/Mo. The results were obtained as follows:

(1) At the loading condition of 23 MW/m^2 for 30 s (the peak temperature was around 800 °C), cracks were not observed on the CVD-W surface even after 300 shots. But in the PM-W, very thin cracks were observed at the loaded area on the surface after 300 shots.
(2) At the loading condition of 50 MW/m^2 for 30 s (the peak temperature was around 1300 °C), cracks due to intergranular fracture were observed at the loaded area of both CVD-W/Mo and PM-W after 50 shots. Especially, a large crack was observed at the loaded area on the surface of the PM-W after 150 shots. The results indicate that the high purity CVD-W is superior to the PM-W in the mechanical properties after recrystallization.
(3) At the loading condition of 100 MW/m^2 for 30 s (the peak temperature was around 1800 °C), a crack due to intergranular fracture was formed in the surrounding area of the loaded area (<1800 °C) on the CVD-W/Mo between the 51st and 75th shot. Because the thermal stresses are released by plastic deformation at the loaded area at around 1800 °C, cracks were easily formed by thermal stresses in the temperature range between 1300 and 1800 °C.

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