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High heat load properties of TiC dispersed Mo alloys

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Abstract

A high heat load experiment of TiC dispersed Mo alloys using electron beams was carried out to investigate fundamental properties of thermal process due to high heat load as high heat flux materials such as the divertor tiles. As a result, although characteristic damage and gas emission of the TiC dispersed Mo alloys around the melting point are inferior to the PM-Mo, but those of the Mo–0.5wt%TiC and Mo–1.0wt%TiC below a temperature of about 2000°C were superior to polycrystalline Mo. Thus, their materials are expected to be suitable as high heat flux materials used below the recrystallization temperature. Results also indicate that it is necessary to release internal strain energy induced by the manufacturing process in order to decrease damage by grain growth and crack formation at high temperature, which was caused by migration of grain boundary and stress, respectively. Characterization of microstructure produced by the rolling process during manufacture is required to design improvement of the thermal conductivity.

Keywords: High-Z wall material; Physical erosion; Impurity source

1. Introduction

Although the utilization of low-Z materials like C/C composite for surface facing components of high heat flux materials such as divertor tiles has enabled the improvement in plasma confinement, their high erosion rates due to sputtering, chemical reaction and radiation enhanced sublimation is now a serious concern as well as due to slow transients and disruptions. Degradation of thermal conductivity by neutron irradiation and high tritium retention could be serious problems in the next generation of D–T fusion experimental reactors [1]. High-Z refractory metals such as tungsten and molybdenum have potential as alternative candidate high heat flux materials owing to their low hydrogen isotope retention and low erosion [2].

However, the grain boundary of group VIa such as tungsten and molybdenum is intrinsically weak due to electron bonding structure [3]. It has been reported that polycrystalline Mo heated to recrystallization temperature becomes brittle and fractures along grain boundaries were formed due to recrystallization embrittlement. This reduced the effective thermal conductivity and hence increased the surface temperature and melting and evaporation due to the heat load from plasma occurred [4,5]. In addition, degradation of mechanical properties such as increase of DBTT (Ductile Brittle Transition Temperature) by neutron irradiation is another important problem [6]. It is therefore necessary to develop the high-Z metal that is not susceptible to either recrystallization embrittlement or irradiation embrittlement.

Regarding recrystallization embrittlement, addition of elements to the high-Z metals is expected to be one of candidate method to increase the recrystallization temperature. In the case of molybdenum, for example, a Mo alloy having small additions of other elements such as TZM (Mo–0.5Ti–0.1Zr) have developed. Recently, high ductile TiC dispersed Mo alloys which have higher recrystallization temperature than that of TZM have been developed by

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Kurishita et al. [7,8]. The alloys have a fine grain size and fine TiC particles were dispersed along the grain boundaries. It is expected that the TiC particles cause strengthening of the grain boundary and hinder the migration of the grain boundary at high temperature. Furthermore, fine grain boundaries will act as sinks for neutron irradiation induced defects as wall as an interface between the TiC and molybdenum matrix. It has been reported that the alloy is superior to powder metallurgy Mo and TZM from the point of view of mechanical behavior [7,8]. In the present study, high heat load experiment of these alloys using electron beams was carried out to evaluate the heat load resistance as high heat flux materials such as divertor tiles.

2. Experimental

2.1. Materials and specimens

The materials were produced by mechanical alloying (MA) at about 1580 K at in an argon atmosphere using a powder of pure Mo, TiC and graphite and sintered by hot isostatic pressing (HIP) treatment at around 1500 K and then hot and warm rolled to 1 mm thickness [7,8]. Compositions of materials used in the present experiment were Mo–0.1wt%TiC, Mo–0.5wt%TiC and Mo–1.0wt%TiC. The materials were cut into 8 mm × 8 mm pieces and prepared for high heat flux experiments. The sample surfaces were mechanically and electronically polished. The samples were also mechanically polished to 0.1 mm thickness and cut into a disk with a diameter of 3 mm ϕ and electropolished to thin foil in preparation for TEM observation. Powder metallurgy Mo (PM-Mo) was also used as a test specimen.

2.2. Experimental device and conditions

The facility used in this experiment was an electron beam irradiation test simulator of the Research Institute for Applied Mechanics of Kyushu University [9,10]. Details of the device have been described elsewhere [9]. The specimens were mechanically fixed on copper block actively cooled with water. The surface temperature of the center region of the sample of diameter 1 mm was measured with a two-color optical pyrometer (1000–3100°C). The two dimensional distribution of the surface temperature was also measured with a thermoviewer, which is a kind of scanning optical pyrometer. The gases emitted from the heated sample surface were detected with a quadrupole mass spectrometer (QMS). The electron beam energy was 20 keV, the power was 28 MW/m^2 (I) and 45 MW/m^2 (II) and duration was 30 s. The beam diameter was about 5 mm. Before and after the irradiation, the sample surface was observed using a scanning electron microscope (SEM). Microstructure of the TiC dispersed Mo alloys was also observed using a transmission electron microscope (TEM).

3. Results

3.1. Microstructure of TiC dispersed Mo alloys

TEM bright field image of the Mo–1.0wt%TiC, as shown in Fig. 1, shows that the characteristic of microstructure is a very fine-grained and a diameter of grain size is around a few 100 nm. Fine TiC particles with a diameter of 20 nm ~ 30 nm are dispersed at grain boundaries as shown in Fig. 2(a). In the case of the Mo–1.0wt%TiC, TiC particles also exists at inside of grain as shown in Fig. 2(b). Grain boundaries as wall as the interface between the TiC particle and Mo matrix are expected to become sinks for radiation defects, because they can act a sink for migrating point defects such as interstitials and vacancies, which are induced by neutron irradiation [11]. Therefore, radiation embrittlement is expected to be suppressed because accumulation of defect is small due to existence of many fine gains and the interface.

3.2. Electron beam irradiation test (I)

Repeated heat flux tests showed that the surface temperature increase was repeatable during the electron beam irradiation, but condition of the contact of the specimen and the copper block surface was changed by exchange of the specimens and the surface temperature changed. Accordingly, the results will be presented by arrange as a function of temperature not heat flux.

SEM images of the samples irradiated are shown in Fig. 3. In the case of the PM-Mo, when surface peak temperature was about 1400°C, the crystal grains grew and small cracks were formed along grain boundaries and the surface became also uneven. The surface was modified and cracks became deep increasing the surface peak temperature. On the other hand, in the case of the Mo–0.5wt%TiC,

Fig. 1. TEM bright field image of Mo–1.0wt%TiC at low magnification.

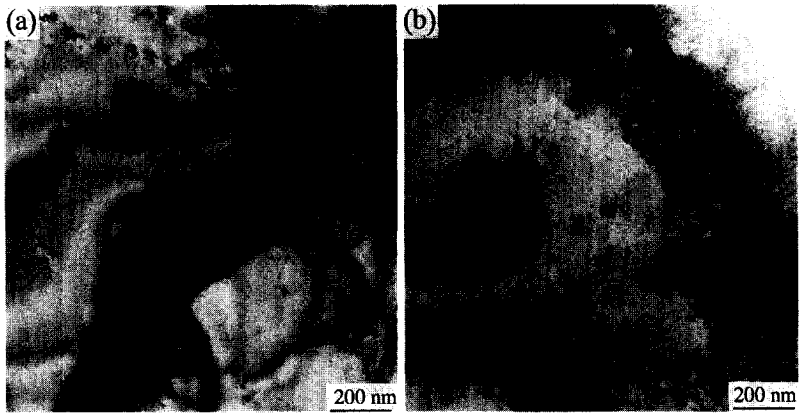

Fig. 2. TEM bright field images of Mo–1.0wt%TiC at high magnification. (a) and (b) show area of grain boundary and inside of grain, respectively.

grain grew, but cracks along grain boundaries and surface modification were not observed. In the case of the Mo–1.0wt%TiC, the surface was slightly modified to become uneven, but large grain growth was not apparently observed. In the case of the Mo–0.1wt%TiC, however, formation of circular damage locally occurred as shown in Fig. 4(a). The enlarged SEM image at the center area shows formation of cracks along the grains and uneven surface as shown in Fig. 4(b). It can be explained that this damage was caused by nonuniformity of the sample. It is necessary to improve the process of manufacture of the materials.

QMS spectra during the electron beam irradiation for the four kind of samples are shown in Fig. 5. Emitted gases were mainly H_2, H_2O, CO and CO_2 irrespective of the sample. These results indicate that vacuum behavior in this temperature range is almost the same as that of the PM-Mo.

3.3. Electron beam irradiation test (II)

Fig. 6 shows SEM images of the three kinds of the samples after the irradiation by 45 MW/m^2 for 30 s, for

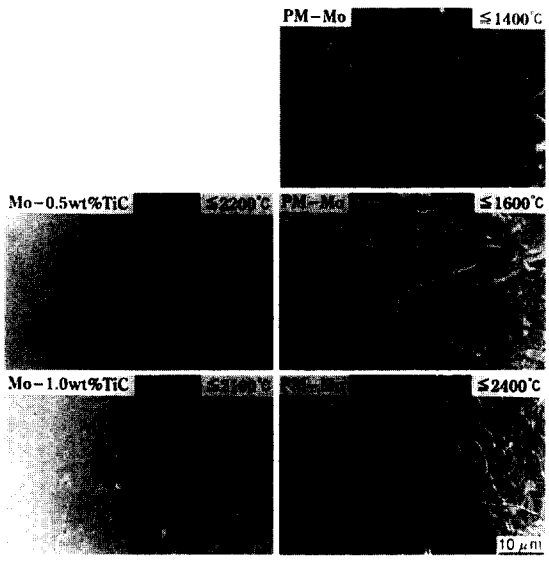

Fig. 3. SEM images of PM-Mo, Mo–0.5wt%TiC and Mo–1.0wt%TiC after electron beam irradiation. Sample and peak surface temperature of center area are indicated in the images.

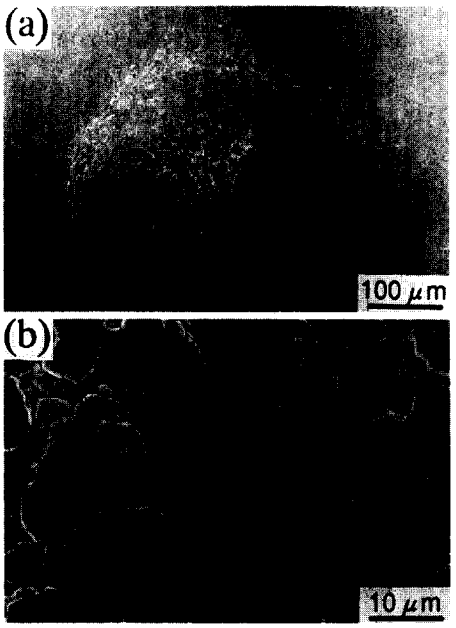

Fig. 4. SEM images of Mo–0.1wt%TiC after electron beam irradiation. Heat flux was 28 MW/m^2 and duration was 30 s. (b) is expanded image of center of area (a).

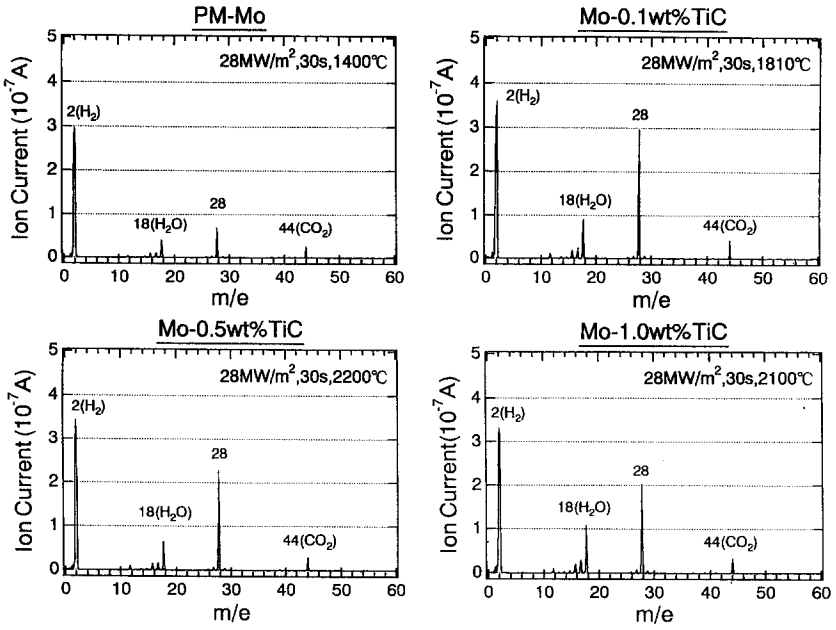

Fig. 5. QMS spectra during electron beam irradiation for (a) PM-Mo, (b) Mo–0.1wt%TiC, (c) Mo–0.5wt%TiC and (d) Mo–1.0wt%TiC. Heat flux is 28 MW/m^2.

which the center area reached a peak surface temperature above the melting point. In the case of the PM-Mo, the irradiated area melted and resolidified and the crystal grains grew. In the outer part of the molten area, grains grew and shallow cracks were formed. On the contrary, in the case of Mo–0.5wt%TiC, deep cracks formed along the grain boundaries as shown in Fig. 6(b)-(2). In the case of the Mo–1.0wt%TiC, thick deep cracks were observed in the melted and resolidified area. The resolidified structure was formed of free grains with a diameter of a few μm. In the outer part of the melted area, grains grew and cracks were formed as shown in Fig. 6(c)-(2).

Fig. 7 shows QMS spectra during electron beam irradiation. In comparison with the experiment (I), large quan-

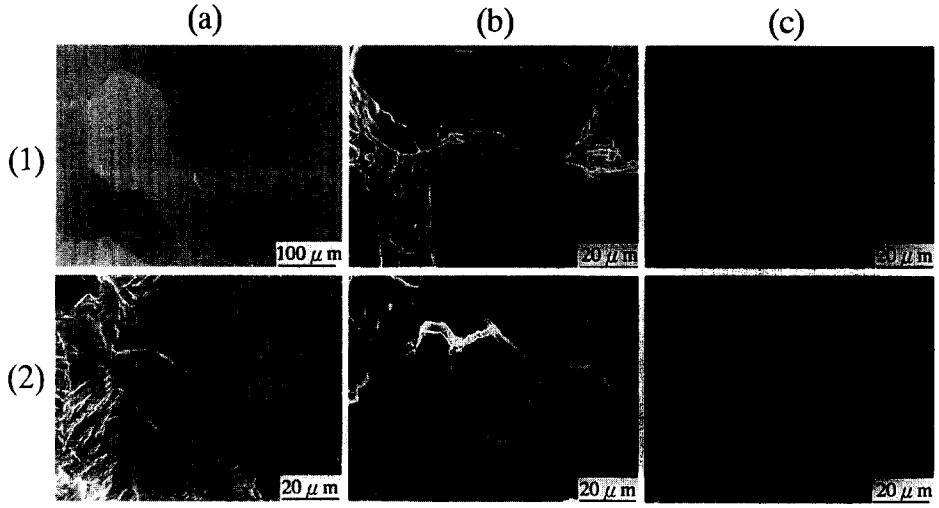

Fig. 6. SEM images of (a) PM-Mo, (b) Mo–0.5wt%TiC and (c) Mo–1.0wt%TiC after electron beam irradiation. (1) Show center area of sample and (2) show outer area of (1). Heat flux is 45 MW/m^2 and duration is 30 s.

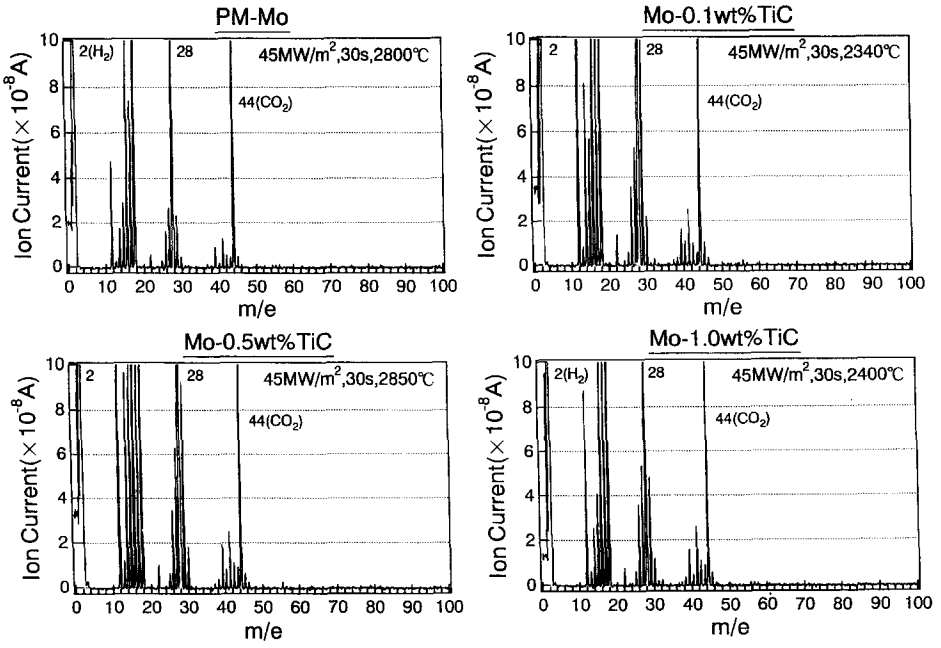

Fig. 7. QMS spectra during electron beam irradiation for (a) PM-Mo, (b) Mo–0.1wt%TiC, (c) Mo–0.5wt%TiC and (d) Mo–1.0wt%TiC. Heat flux is 45 MW/m^2.

tity of gases were emitted. The amount of hydrocarbons and C from the TiC dispersed Mo alloys was larger than that of the PM-Mo. These results indicate that the behavior of gas emission of the TiC dispersed Mo alloys is inferior to that of the PM-Mo in the temperature range around the melting point.

4. Discussion

Although damage and gas emission of the TiC dispersed Mo alloys around the melting point are inferior to those of PM-Mo, those of Mo–0.5wt%TiC and Mo–1.0wt%TiC below temperature of about 2000°C are superior to the PM-Mo. Thus, these materials are expected to be suitable as high heat flux materials used below the recrystallization temperature, which is higher than that of the PM-Mo.

The internal strain energy in the grain of the TiC dispersed Mo alloys is expected to be considerable higher than that of the PM-Mo, because a mechanical alloying treatment was used during the manufacture process. Release of the internal strain energy at high temperatures causes the migration of grain and formation of stress, which lead grain growth and cracking, respectively. Accordingly, when pinning effect of the TiC particle for the migration of the grain boundary (namely, TiC particle act as obstacle for the grain growth) becomes to be weak and/or energy of grain growth becomes strong, deeper and longer cracks and larger grains than for the PM-Mo are expected to be formed because of the release of strong internal strain energy at high temperature. Below the recrystallization temperatures of the TiC dispersed Mo alloys, which is higher than that of the PM-Mo, the TiC dispersed Mo alloys are superior to the PM-Mo. On the other hand, above the recrystallization temperature, they are inferior to those of the PM-Mo because they have high internal strain energy in the grain. These results indicate that it is necessary to release internal stress during the manufacture process. Further study is required to investigate the release condition of the stress.

The surface temperature depends on the thermal conductivity of materials in actively cooled system in steady state. Therefore, even if the recrystallization temperature is high, it is necessary that the thermal conductivity is high enough. Measurement of the thermal conductivity in the direction perpendicular to the roll process by a laser flash method showed that of the thermal conductivity of the TiC dispersed Mo alloys was decreased to about 0.76 times that of the PM-Mo [12]. The thermal conductivity after the HIP treatment was the same as that of the PM-Mo irrespective of content of the TiC. These results indicate that the decrease of the thermal conductivity of the TiC dispersed Mo alloys could not be attributed to TiC particles, but to the structure which was formed by the roll process following the HIP treatment. Characterization of the microstruc-

ture produced by the rolling process is required to design improvements of the thermal conductivity.

In the present study, small size specimens were used to investigate fundamental properties of thermal process due to high heat load. Further study would be required to evaluate large size materials, which is used in the fusion devices.

5. Conclusions

High heat load experiments of TiC dispersed Mo alloys using electron beams were carried out to investigate fundamental properties of thermal process due to high heat load as high heat flux materials such as the divertor tiles. As a result, although characteristic damage and gas emission of the TiC dispersed Mo alloys around the melting point are inferior to the PM-Mo, those of Mo–0.5wt%TiC and Mo–1.0wt%TiC below temperature of about 2000°C were superior to polycrystalline Mo. Thus, these materials are expected to be suited to be high heat flux materials used below recrystallization temperature. Results also indicate that it is necessary to release the internal strain energy induced by manufacture process in order to decrease damage by grain growth and crack formation at high temperature, which was caused by migration of grain boundary and stress, respectively. Characterization of the microstructure produced by roll process during manufacture is required to design improvement of the thermal conductivity.

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