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Plasma material interaction studies on low activation materials used for plasma facing or blanket component

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Abstract

Numerous issues on the plasma material interactions were investigated for low activation materials. Co-deposited carbon dust was prepared and the deuterium concentration was measured. The concentration was approximately half of the present design value for ITER. For ferritic steel, the deuterium retention was observed to be comparable to that of stainless steel. Physical sputtering yield was roughly the same as that for stainless steel. For the reduction of absorption rate in vanadium alloy, titanium oxide coating was conducted, and the coating was observed to be very effective for reduction of hydrogen absorption. Helium gas permeability was measured for numerous SiC/SiC composites, and the SiC/SiC composite made by the NITE process showed quite low permeability. The SiC/SiC blanket may be able to be used without helium leakage into plasma.

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1. Introduction

In the fusion experimental reactor, ITER, C/C composite (graphite) and tungsten are used as materials of divertor plate [1]. The C/C composite is employed for divertor target regions. The shield blanket modules are made of stainless steel. Graphite and tungsten are low activation materials but stainless steel is not. After starting machine operation, the graphite is significantly eroded by high particle and heat fluxes, and the carbon dust is produced. It is indicated that the carbon dust has a large tritium inventory. However, there is no systematic study on estimation of fuel hydrogen retention in carbon dust. The evaluation of fuel hydrogen retention in carbon dust has been recognized as an important issue in the design of ITER. In the present study, the fuel hydrogen inventory of carbon dust is evaluated based upon the experimental studies of deuterium retention in carbon dust [2,3].

In fusion demonstration reactors, the blanket module will be made of low activation materials such as ferritic steels, vanadium alloys and SiC/SiC composites [4,5]. The first wall of blankets made of these materials is directly exposed to the plasma environment, if an armor layer is not used. Thus, issues associated with plasma material interactions have to be investigated [4]. There are concerns with respect to gas desorption, sputtering and fuel hydrogen inventory for ferritic steels [6]. For vanadium alloys, the possibility of hydrogen embrittlement has been suggested [7]. In the case of blanket made of SiC/SiC composites, there is a concern for permeation of helium gas coolant into the plasma [8]. The problems associated with these issues were studied

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at Hokkaido University. These results are described in this paper.

2. Plasma material interactions of low activation materials

2.1. Fuel hydrogen retention in carbon dust

In ITER, the divertor wall materials are C/C composite for the region of the divertor target and tungsten for other divertor regions. Eroded carbon deposits on the wall in the form of carbon dust. The major process for the production of carbon dust is the erosion of the C/C composite during the disruptions. It is regarded that the carbon dust has a large inventory of fuel hydrogen. If the in-vessel tritium inventory exceeds 450 g, a periodic cleaning is conducted for reduction and recovery of tritium inventory of ITER plant. If the cleaning has to be done often, the plasma operation time becomes to be significantly limited. There is little data on estimation of tritium retention in carbon dust. In the present study, a systematic evaluation of tritium retention in carbon dust was carried out.

Carbon dust deposited on the shadow region from core plasma will be exposed to molecular hydrogen. A question is the amount retained in the dust by this process. For carbon dust prepared by electron beam evaporation, a hydrogen gas absorption experiment was conducted by varying the temperature of the dust and the deuterium gas pressure. Under the ITER condition with temperature of 300 °C and pressure of 1 Pa, the absorption amount was observed to be very small, of order of 10^{-3} in the atomic ratio of D/C. The carbon dust in the vicinity of the divertor target is exposed to fuel hydrogen ions. Hence, a deuterium ion irradiation experiment was conducted using an ECR ion irradiation device [2]. The retained amount was very similar to the experimental results for graphite. When the wall temperature at the divertor is higher than approximately 700 °C, the retained amount becomes very small, lower than 0.1 in the atomic ratio of D/C. The eroded carbon also deposits on the wall with fuel hydrogen. This dust is called co-deposited carbon dust. The co-deposited carbon dust was prepared by using an arc discharge, where the discharge gas was deuterium and the electrodes graphite. In this apparatus, the carbon dust co-deposited with deuterium on the sample. During the deposition, the sample temperature was 380 °C and the gas pressure was varied from 1 to 15 Pa. The deuterium concentration was measured using a technique of thermal desorption, TDS. Fig. 1 shows the atomic ratio of D/C versus deuterium gas pressure. At a pressure of 1 Pa, the atomic ratio of D/C was approximately 0.2. This value is a half of that measured in the ion irradiation experiment, D/C = 0.4 at room temperature, RT. For the case



Fig. 1. Deuterium concentration in atomic ratio of D/C of codeposited carbon dust versus deuterium gas pressure in arc discharge.

of ITER, the hydrogen concentration of the carbon dust has been assumed as the value at RT, 0.4. The present result suggests that the cleaning period for reduction of in-vessel tritium inventory may be twice as long as estimated period based on the ion irradiation experimental data at RT.

2.2. Gas desorption, physical sputtering and fuel hydrogen retention of ferritic steel

Ferritic steel, F82H, has been developed by Japan Atomic Energy Research Institute, JAERI, as the blanket structure material of fusion demonstration reactors. This material, however, is oxidized faster than stainless steel. Hence, there is a concern for desorption of gases containing oxygen. A gas desorption experiment was conducted for F82H exposed to the open air atmosphere at RT for 2 years. The desorption amounts of CO and H₂O were significantly larger than those for stainless steel, SS 316L. However, the gas desorption amount of F82H was effectively reduced to the level of SS 316L after pre-baking at 300 °C. Even after the prebaking, the desorption of CO was relatively large although the desorption of H_2 was relatively large in the case of SS 316L. In a fusion demonstration reactor, ferritic steel may be used as the first wall of the blanket and other vessel components. In such case, there is a concern for physical sputtering owing to charge exchanged neutrals and ions. In order to obtain the sputtering yield, a deuterium ion irradiation experiment was conducted for F82H, using an ECR ion source. After the irradiation, the sputtering yield was obtained from the weight loss. For 1.7 keV deuterium ion, the sputtering yield was 0.04 atom/ion, which is roughly the same for SS 316L.



Fig. 2. Deuterium concentration at RT of unpolished ferritic steel, mechanically polished ferritic steel, stainless steel and graphite in atomic ratio of D/C versus deuterium ion fluence.

The deuterium retention of F82H was also investigated by deuterium ion irradiation at RT, followed by TDS analysis. In F82H with a thick oxide layer (60 nm), the retained amount of deuterium remained roughly the same even when the ion fluence increased. In F82H with a thin oxide layer (15 nm), the retained amount increased with the ion fluence. In both cases, the retained amount became comparable when the fluence was high, as shown in Fig. 2. In the F82H with a thick oxide layer, it was seen that the oxide layer was thinned by ion sputtering, similar to the case of F82H with a thin oxide layer. In the F82H with a thick oxide layer, the deuterium might have been well trapped in the oxide layer when the fluence was low. This is a possible reason for the difference in the fluence dependence. The retained amount was compared with results on SS 316L and graphite. The retained amount in F82H was roughly the same as that in SS 316L and one order of magnitude smaller than that in graphite. The retained deuterium in F82H desorbed at a relatively low temperature regime, 400 °C. Then, the temperature required for reduction of fuel hydrogen retention becomes lower, compared to the case of graphite, 800 °C.

The above experimental results show that the use of ferritic steel does not cause the additional problems associated with plasma material interactions, compared to the case of SS 316L.

2.3. Hydrogen absorption of vanadium alloy

V-4Cr-4Ti alloy is a candidate material for blanket structure. This vanadium alloy is exposed to environment containing hydrogen gas or ion. It is known that vanadium metal and its alloy easily absorbs hydrogen and that hydrogen embrittlement takes place if the hydrogen concentration exceeds 40–100 wppm [7]. Equilibrium concentration data show that the concentration exceeds the critical concentration when the temperature is lower than approximately 300-400 °C. Although the operating temperature of a vanadium alloy blanket is 600 °C, in the starting and shutdown phases the temperature decrease will drop below 300-400 °C. Thus, the concentration may exceed the critical concentration in such the environment. It is necessary to develop a method to avoid the hydrogen absorption. It is known that Ti-O coatings on vanadium alloys reduces the hydrogen absorption rate since the diffusion rate of hydrogen in Ti-O is very much smaller than that in vanadium alloys [7]. Based on this principle, the absorption amount of hydrogen in a Ti-O coated vanadium alloy was measured, and compared with a non-coated vanadium alloy. The Ti-O coating was deposited on entire surface of the vanadium using a magnetron sputtering apparatus. The thickness of Ti-O coating was in the range from 0.13 to 1.1 µm. The Ti-O coated sample was placed in the vacuum chamber with a hydrogen pressure of 40 Pa, and the sample temperature was kept at 300 °C. The absorption amount was measured by the reduction of hydrogen gas pressure. Fig. 3 shows the change of hydrogen gas pressure versus absorption time. The data for the non-coated sample is also shown in this figure. The reduced amount of the pressure corresponds to the absorption amount. In the case of the non-coated sample, the absorption amount corresponded to a hydrogen concentration of approximately 60 wppm, which is roughly the same as the equilibrium concentration. With an increase of the coating thickness, the absorption speed significantly decreased. The reduction of absorption rate in the sample with a coating thickness larger than 0.52 µm was one or two orders of magnitude smaller than that in the non-coated sample. In order to evaluate the thermal stability of Ti-O coating, the Ti-O coated sample was



Fig. 3. Change of deuterium gas pressure versus absorption time for Ti–O coated vanadium alloy and non-coated vanadium alloy.

heated up to 700 °C and held for 5 h. In the coating layer, significant diffusion of Ti and O was not observed in a temperature range below 600 °C. These results suggest that the Ti–O coated vanadium alloy can be used for a long time period even with the temperature in the startup and shutdown phases. For the practical use, the insertion of Ti–O layer in the component of vanadium alloy may be suitable compared with the case of coating.

2.4. Permeation of helium gas through SiC/SiC composite

SiC/SiC composites have been very rapidly developed as candidate blanket materials for fusion demonstration reactors [5]. Since the operation temperature can be high, approximately 800 °C, a high energy conversion efficiency is expected. In the SiC/SiC blanket, helium gas is used as a coolant. Hence, a major concern is helium gas permeation through the SiC/SiC coolant piping and first wall into the plasma. There are few permeation experiments of helium gas for SiC/SiC composite. In order to evaluate the potentials of using SiC/SiC composite as blanket material, experiments of helium gas permeability for numerous SiC/SiC composite materials were conducted. The apparatus for measurement of helium permeability was assembled [9]. The apparatus consists of high pressure and low pressure chambers. The sample with a size of $15 \times 15 \times 2$ mm was placed between two chambers, with epoxy resin used to form vacuum seals. Fig. 4 shows the helium gas permeability in m²/s versus helium gas pressure of the high pressure chamber for numerous SiC/SiC composite samples. The permeability is defined by $K = (P_L/P_H)(D/A) S_{eff}$, where $P_{\rm H}$ and $P_{\rm L}$ are helium gas pressures in high and low pressure chambers, respectively, D and A thickness and area of the sample, respectively, and Seff effective pumping speed at the low pressure chamber. The samples, PIP, PIP + MI, HP, LPS1 and LPS2, are SiC/SiC



Fig. 4. Helium gas permeability measured at RT versus helium gas pressure of high pressure chamber for numerous SiC/SiC composites.

composites made by polymer infiltration pyrolysis, polymer infiltration pyrolysis plus melt infiltration, hot pressing, liquid phase sintering plus hot pressing using sub-micron powder β -SiC, and liquid phase sintering plus hot pressing using nano-powder β -SiC, respectively. LPS3 is a bulk SiC sample made by liquid phase sintering plus hot pressing using nano-powder β -SiC. The production method for LPS samples is called NITE process [5]. The observation of surface morphologies for these samples using a scanning electron microscope, SEM, showed that LPS3, LPS2 and LPS1 had quite dense structures compared with the other samples. The permeability for LPS3, LPS2 or LPS1 was very small compared with those of PIP, PIP+MI and HP. This result roughly corresponds to the porous structure observed by SEM. The lowest permeability as SiC/SiC composite was observed in LPS2. The value was only of order of 10^{-11} m²/s, which is small enough to avoid the leakage of helium gas into the plasma [9]. For the PIP sample, a thermal cycling experiment was carried out and then the permeability was measured. In the experiment, the heating temperature was cycled to 800 °C for 60 cycles. The permeability remained the same even after the heat cycle experiment. This result shows that the permeability may not increase during the operating life of the SiC/SiC blanket. Further heat cycle experiment for LPS samples will be conducted.

3. Discussion and summary

In the present study, several problems associated with plasma material interactions with low activation materials were pointed out, and experimental results were briefly described. In ITER, one major concern is reduction of in-vessel tritium inventory in carbon dust. The co-deposited carbon dust was prepared using a deuterium arc discharge, by changing the deuterium gas pressure. The deuterium retention was measured by a TDS technique. The deuterium concentration in the atomic ratio of D/C was approximately 0.2, which is a half of the design value. Thus, this result suggests that the time period for planned cleaning can be doubled.

Ferritic steels are regarded as the most promising low activation material. There are concerns on fuel hydrogen retention, gas desorption and sputtering yield. Experiments on these issues showed that the influence of ferritic steel on plasma was very similar to that of stainless steel. Vanadium alloys have been now rapidly developed. In the case of vanadium metal or alloy, a major concern is hydrogen embrittlement owing to large hydrogen absorption. The embrittlement takes place when the temperature is lower than 300–400 °C. In the startup and shut down phases of a vanadium alloy blanket, the temperature may drop to this regime. Thus, it is required to develop the method to avoid the hydrogen absorption. The effect of Ti–O coating was studied for the vanadium alloy. By the Ti–O coating, the hydrogen absorption rate was significantly reduced. The application of this scheme may contribute to lengthen the lifetime of vanadium alloys. SiC/SiC composites have also been developed as blanket material. Since SiC/ SiC is a ceramics, there is a concern on leakage of helium coolant into the plasma. For numerous SiC/SiC composites, the helium gas permeability was measured. The SiC/SiC composite made by the NITE process showed quite low permeability, only 10^{-11} m²/s. If this value can be achieved under the operation conditions, the leakage of helium coolant into the burning plasma can be avoided.

These results show that present low activation materials may be used as plasma facing components, and the data obtained are useful for the designs of ITER and fusion demonstration reactors.

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References

- IAEA, Technical basis for ITER-FEAT outline design, ITER EDA Documentation Series No. 19, IAEA, Vienna, 2000.
- [2] T. Hino, H. Yoshida, Y. Yamauchi, Y. Hirohata, K. Nakamura, M. Akiba, Fus. Eng. Des. 61&62 (2002) 605.
- [3] H. Yoshida, Y. Hirohata, Y. Yamauchi, K. Yokoyama, Y. Hirohata, M. Akiba, T. Hino, Fus. Sci. Technol. 41 (2002) 943.
- [4] T. Hino, Y. Hirohata, Y. Yamauchi, S. Sengoku, in: Proceedings of 18th IAEA Fusion Energy Conference, IAEA-CN-77/FTP1/08, IAEA, 2000.
- [5] A. Kohyama, Y. Katoh, S.M. Dong, T. Hino, in: Proceedings of 19th IAEA Fusion Energy Conference, IAEA-CN-77/FT/P1-02, IAEA, 2002.
- [6] T. Hino, K. Yamaguchi, Y. Yamauchi, Y. Hirohata, K. Tsuzuki, Y. Kusama, in: Fourth General Scientific Assembly of Asia Plasma Fusion Association on New Development of Plasma Physics and Fusion Technology, Hangzhou, China, 13–16 October 2003.
- [7] Y. Hirohata, D. Motojima, T. Hino, S. Sengoku, J. Nucl. Mater. 313–316 (2003) 172.
- [8] T. Hino, T. Jinushi, Y. Yamauchi, M. Hashiba, Y. Katoh, A. Kohyama, Ceram. Trans. 144 (2003) 353.
- [9] T. Hino, T. Jinushi, Y. Hirohata, M. Hashiba, Y. Katoh, A. Kohyama, Fus. Sci. Technol. 43 (2003) 184.