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Development of Thermal Desorption Spectrometer for Materials Irradiated with Neutrons

中性子照射材料における昇温脱離ガス分析法の開発

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Neutron irradiation will modify the behavior of the plasma material interactions (the PMIs) substantially. An instrument for the thermal desorption spectroscopy (the TDS) on neutron irradiated materials was developed for study of the PMIs. The instrument is composed of a high-vacuum chamber, and two quadrupole mass-spectrometers, one having a conventional mass resolution with a mass range up to 100 amu, and the other having a high resolution of being able to separate D_2+ and He+, with a mass range up to 6 amu. The instrument is also facilitated with a low energy light ion gun. The system is installed in the hot laboratory of the Oarai Branch of the Institute for Materials Research of Tohoku University, where a variety of radio-isotopes could be handled. With this instrument, desorption behaviors of nuclear transmuted helium and deuterium were measured on the neutron irradiated ceramics. The clear desorption spectra of the helium and the deuterium were obtained on the silicon nitride (Si_3N_4). The desorption behavior of the transmuted helium and deuterium is definitely different from that of surface-implanted ones. The results are demonstrating that the present system could analyze the complicated dynamics of desorption behavior of the mixed helium and deuterium atoms injected into neutron irradiated materials.

1. Introduction

Neutron irradiation will modify the behavior of the plasma material interactions (the PMIs) substantially. The instrument for the thermal desorption spectroscopy (the TDS) on neutron irradiated materials was developed for study of the PMIs. The instrument is composed of a high-vacuum chamber, and two quadra-pole mass-spectrometers, one having a conventional mass resolution with a mass range up to 100 amu, and the other having a high sensitivity and high resolution of being able to separate D_2+ and He+, with a mass range up to 6 amu. The instrument is also facilitated with a low energy light ion gun.

2. Experimental

The system is installed in the hot laboratory of the Oarai Branch of the Institute for Materials Research of Tohoku University, where a variety of radio-isotopes could be handled[1]. With this instrument, desorption behaviors of nuclear transmuted helium and deuterium were measured on the neutron irradiated ceramics as well as on the ion-planted ceramics which were irradiated in the fission reactors. The neutron irradiations were carried out in the fast reactor JOYO and the mixed spectrum reactor JMTR in the Oarai Research Establishment of the Japan Atomic Energy Agency.

3. Result and discussion

The clear desorption spectra of the helium and the deuterium were obtained on the silicon nitride (Si_3N_4) irradiated in the JOYO up to $5.5x10^{26}n/m^2$ at 710C as shown in Fig. 1.

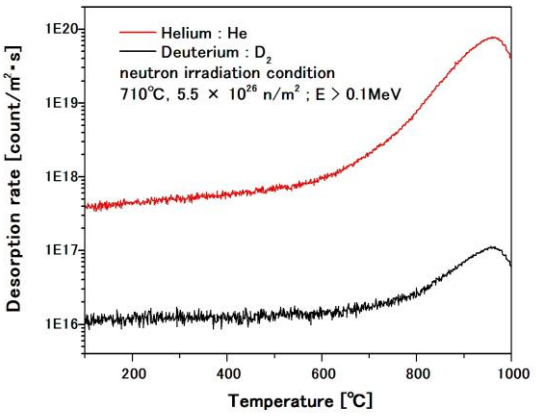

Fig.1 desorption rate of the helium and the deuterium of the silicon nitride irradiated in the JOYO.

The proton formation by the thermal neutron

through the $^{14}N(n,p)^{14}C$ nuclear transmutation is well-known but the production of deuterium and helium was not expected[2]. It is assumed that the neutrons with their energy higher than 1 MeV would have contributed to the formation of the helium and the deuterium. The experimentally obtained results on the amount of the generated gases would be analyzed by comparing with the calculated estimation. The desorption behavior of the transmuted helium and deuterium is definitely different from that of surface-implanted ones. Fig. 2 shows the desorption spectrum from the irradiated Si_3N_4 and the 3 keV helium implanted.

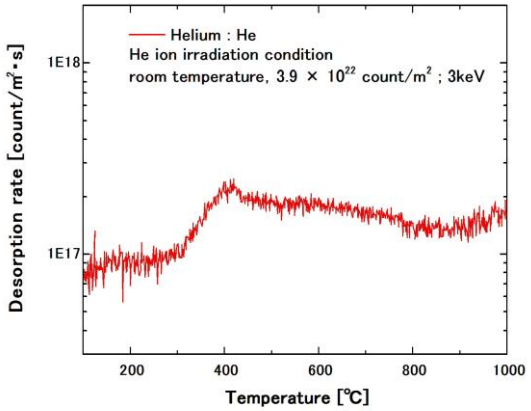

Fig.2 The desorption rate of the helium from the silicon nitride which implanted 3 keV helium ion

The multiple and much broader desorption peaks are observed generally at lower temperatures, suggesting that the trappings are more shallow and there are a variety of trapping sites with a variety of trapping energies. Fig. 3 shows the microstructure of the neutron irradiated Si_3N_4 up to $5.5x10^{26}$ n/m^2 (E>0.1MeV).

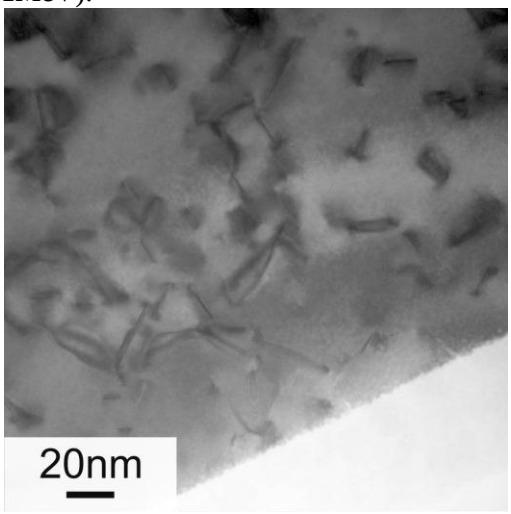

Fig.3 the microstructure of the neutron irradiated Si_3N_4 up to $5.5x10^{26}$ n/m^2 (E>0.1MeV).

Only the dislocation loops were observed and they were analyzed to be the interstitial type dislocation loop which has the Burgers vector of $1/6\langle 023\rangle$, inserted into the $\{10\bar{1}0\}$plane[3]. The evaluation of the fracture toughness of the irradiated Si_3N_4 suggested that some small defects would exist and enhance the crack propagation. The corresponding defects are speculated to be vacancies or vacancy complexes, because when this range of neutron fluence, the swelling of the Si_3N_4 occur[4]. The voids and vacancies contribute to swelling[5].

It is interpreted that the sharp peak at 950C in Fig.1 is corresponding to the detrapping of the helium from the vacancy or vacancy clusters. In the meantime, a small peak could be separated at about 650C. which could be detrapping of the helium from the above mentioned interstitial dislocation loops.

Gas release could not be detected in the highly pure SiO_2 irradiated in the JMTR, but could be detected in the Al_2O_3. Taking other results into consideration, the oxygen would not contribute to the gas nuclear transmutation in the present irradiation. The observed gas release from the Al_2O_3 would be attributed to the impurities-originated gas nuclear transmutation, as the Al_2O_3 contains more impurities than SiO_2.

The results are demonstrating that the present system could analyze the complicated dynamics of desorption behavior of the mixed helium and deuterium atoms injected into neutron irradiated materials

4.Reference

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