

§2. Studies of Interaction between Cooling Pipe Materials and Tritium, and Their Chemical Behavior

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i) Introduction

Stainless Steel (SS-304 [1], 316 etc) is expected to be used in fusion reactors as various component materials like cooling pipe because of its good mechanical properties and corrosion resistance. The behavior of tritium in SS is one of the most important issues for the assessment of fusion safety. However, the chemical behaviors of hydrogen isotopes with various adsorption/absorption have not been well studied. In the present study, the typical material for components, SS-316, was chosen as specimen and deuterium was charged either by water adsorption, ion irradiation or electrolysis. The chemical states of iron, chromium, nickel and oxygen on SS-316 were observed by the XPS. The TDS was also applied to the analyses of the thermal desorption behaviors of D_2 and D_2O from SS-316.

ii) Experimental

The SS-316 sample with size of $10 \times 10 \times 1 \text{ mm}^3$ was used. Two kinds of sample with different surface finish, namely the non-pretreated sample and pretreated sample by mechanical polish and annealing at 1273 K in vacuum for 30 minutes to remove surface oxide layers, have prepared. For these samples, D_2 or D_2O was sorbed on/in the sample by various methods, such as water adsorption, ion irradiation and electrolysis. In the water adsorption, the sample was immersed in heavy water for 30 min. In the electrolysis experiment, the sample was used as a cathode for 60 min with the current of 0.1 A. For deuterium ion irradiation, the 4.0 keV D_2^+ ion was implanted into the sample with the flux of $5.1 \times 10^{18} \text{ D}^+ \text{ m}^{-2} \text{ s}^{-1}$ up to the fluence of $2.04 \times 10^{22} \text{ D}^+ \text{ m}^{-2}$. The chemical states of iron, chromium, nickel, molybdenum, carbon and oxygen on the SS-316 specimen were evaluated by X-ray photoelectron spectroscopy (XPS). The thermal desorption spectroscopy (TDS) was also applied to the evaluation of the desorption behavior of hydrogen isotopes from the stainless steel. The heating rate was set to 30 K/min from room temperature to 1273 K.

iii) Results and discussion

In TDS experiment, it was found that the desorption stage

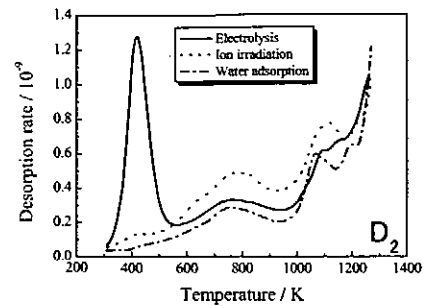


Fig. 1 TDS spectra of deuterium from non-pretreated SS-316 with various treatment

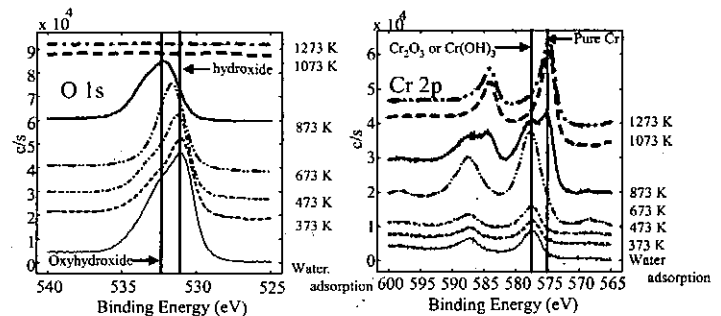


Fig. 2 XPS spectra of O 1s and Cr 2p from non-pretreated SS-316 with water adsorption as a function of heating temperature.

consisted of three stages as shown in Fig. 1. To elucidate the desorption behavior in detail, the sample was annealed isochronally from room temperature to 1273 K and XPS analysis was performed. Figure 2 shows the typical photoelectron spectra of O 1s and Cr 2p of after heating of SS-316 with water adsorption by XPS. It was found that Cr_2O_3 or $Cr(OH)_3$ was a typical chemical form just after water adsorption. However, the chemical form was changed to pure chromium after heating at 873 K. This temperature region corresponds to that of second desorption stage. It was also found that the first desorption stage of TDS was the deuterium desorption formed as oxyhydroxide, namely $FeOOD$ or $CrOOD$, on the surface of the sample. The third desorption temperature was about 1050 K. This temperature corresponds to the temperature just after the decomposition of oxyhydroxide. Therefore, the retained deuterium was desorbed by the decomposition of oxyhydroxide.

iv) Conclusion

The chemical forms of hydrogen isotopes on/in SS-316 were studied by XPS and their desorption behavior was evaluated by TDS. It was found that the deuterium desorption stages consisted of three stages. The first, second and third stages would correspond to the desorption of deuterium by the decomposition of oxyhydroxide and hydroxide, and of retained deuterium absorbed in SS-316, respectively.

References

1) Oya, Y. et al., Fusion Sci. & Tech., **44** (2003) 359.