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Effects of hydrogen atmosphere on mechanical properties and surface conditions of a reduced activation ferritic steel F82H

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Abstract

Effects of hydrogen atmosphere on mechanical properties and surface conditions have been studied for a reduced activation ferritic steel F82H (Fe–8Cr–2W–V–Ta). Test specimens of F82H were exposed to hydrogen gas atmosphere simulating the operating conditions of a fusion reactor. Surface analyses were performed by Auger electron spectroscopy in order to evaluate the depth profiles of surface atomic compositions of F82H, and surface oxidation layers with a thickness of up to 56 nm were observed on the surface, mainly due to the high diffusion rate of oxygen in F82H matrix. A quantitative analyses of the specimens exposed to hydrogen atmosphere were performed by a gaschromatograph, and absorbed hydrogen in F82H was found negligibly small, less than 5 ppm. Tensile and Charpy impact tests were carried out on these specimens, and mechanical properties were found comparable to those of as-received materials.

1. Introduction

A reduced activation ferritic steel, F82H (Fe–8Cr–2W–V–Ta), is one of the candidates for the first wall and the blanket structural materials because of its intrinsic high resistance to void swelling under heavy neutron irradiation [1], and is the primary candidate structural material of the pebble-bed-type solid breeder blanket of Steady State Tokamak Reactor (SSTR) [2] and Japanese test modules to be tested in ITER [3]. This steel is also expected to have better thermal properties compared with those of austenitic stainless steels, although not enough data have been available, especially on the mechanical and vacuum properties needed for the application to fusion reactors as well as heavy neutron irradiation behaviours. As this steel is to be used in ultra high vacuum in the plasma chamber and also under hydrogen atmosphere during plasma operation, vacuum properties such as hydrogen gas absorption characteristics are one of the key issues to be examined for the application of this steel to fusion reactors. This steel has been selected as one of the two reference steels for the IEA round robin test on low activation ferritic steels [4], and extensive efforts have been made to characterize this steel and to accumulate a wide range of material properties [5]. As for the vacuum properties and surface conditions of this steel, gas desorption properties have been reported in [5], and recently outgassing properties have been also reported by means of through-put method [6]. The effects of hydrogen atmosphere on mechanical properties and surface conditions of this steel have not yet been reported. The purpose of this study is to carry out an exposure of F82H steel to hydrogen atmosphere and its effects on the mechanical properties and surface conditions.

2. Experimental procedures

Test specimens used in the present study were machined from a hot-rolled F82H plate, 7.5-mm-thick, taken from a high-purity 5-ton ingot. The geometry of tensile and Charpy impact test specimens is shown in Fig. 1(a) and (b), respectively. Sheet type tensile specimens, 4.0×4.0 mm^2 in cross section, were machined from 7.5 mm thick $\times$ 400 mm $\times$ 300 mm F82H square plates following JIS (Japan Industrial Standard) Z-2201-14B. The longitudinal direction of the tensile specimen was taken in perpendicular to the rolling direction of the

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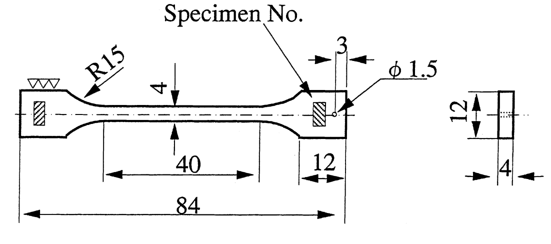
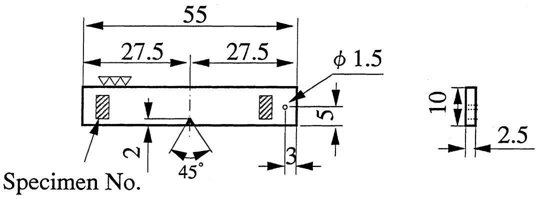


(a) Tensile test specimen
(JIS Z-2201-14B)



(b) Charpy impact test specimen
(JIS Z-2202-4,1/4 size)

Fig. 1. The geometry of tensile and Charpy impact test specimen (dimension in mm).

original plate. Charpy impact test specimens of F82H were quarter-size standard Charpy specimens based on JIS Z-2202-4 with a 2-mm-deep 45V notch (no side groove) and a 0.225–0.275-mm root radius. The longitudinal direction of the Charpy impact test specimens was perpendicular to the rolling direction with a notch along the rolling direction. These specimens were cleaned by acetone in order to remove the surface contamination prior to the following tests. These specimens were utilized for both of the mechanical tests and surface analysis after exposure to hydrogen atmosphere mentioned below.

Test specimens were set in the vacuum chamber made from austenitic stainless steel (SS316) and pre-heated by infrared-ray heaters, and then hydrogen gas was introduced within the vacuum chamber under three different conditions. The parameters of the hydrogen exposure conditions are summarized in Table 1. Exposure condition No. 1, the hydrogen pressure of 10^{-5} Torr and the temperature of 500°C kept for 15 min, meant the flashing or baking treatment. Exposure condition No. 2, the hydrogen pressure of 10^{-2} Torr and the temperature of 300°C kept for an hour, simulated the hydrogen atmosphere anticipated in a fusion reactor. In the condition No. 3, the hydrogen pressure and temperature was raised up to 600 Torr and 500°C, respectively, for 5 h to accelerate interaction of F82H with hydrogen. The same tests were conducted for SS316L with same specimen size for the comparison purpose.

Table 1
Parameters of the hydrogen exposure conditions

No.	Heating duration	Heating temperature	Hydrogen pressure
1	15 min	500°C	$<10^{-5}$ Torr
2	1 h	300°C	10^{-2} Torr
3	5 h	500°C	600 Torr

3. Results and discussion

3.1. Surface analysis

Surface analyses of the as-received F82H specimen and these specimens exposed to hydrogen atmosphere under the conditions listed in Table 1 were performed by Auger electron spectroscopy in order to evaluate the depth profiles of surface atomic compositions. The surfaces of each specimen were etched by an argon ion beam. Auger peak heights obtained were converted into atomic concentrations along the depth from the surface. The results of the as-received F82H and SS316L specimens are shown in Fig. 2(a) and (b), respectively. As is shown in these figures, the surfaces of both the F82H and SS316L specimens were contaminated by carbon compositions and also an oxidation layer was formed. The thickness of the oxidation layers were estimated to be around 7 nm for both materials by defining the thickness of the oxidation layer by the depth where the oxygen concentration was reduced to a half of the peak value.

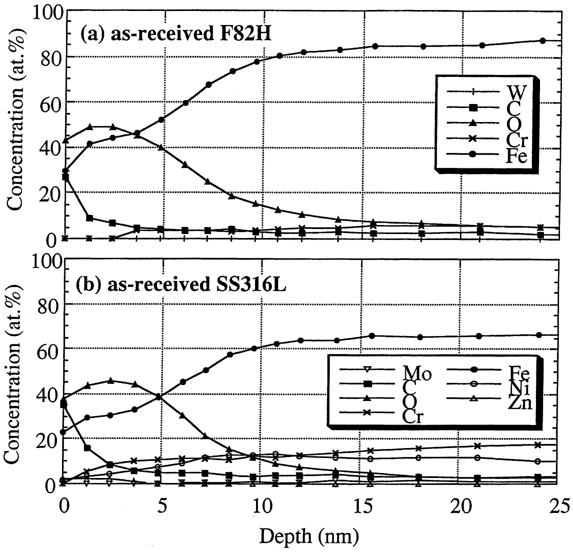


Fig. 2. The depth profiles of surface atomic composition of as-received F82H and SS316L.

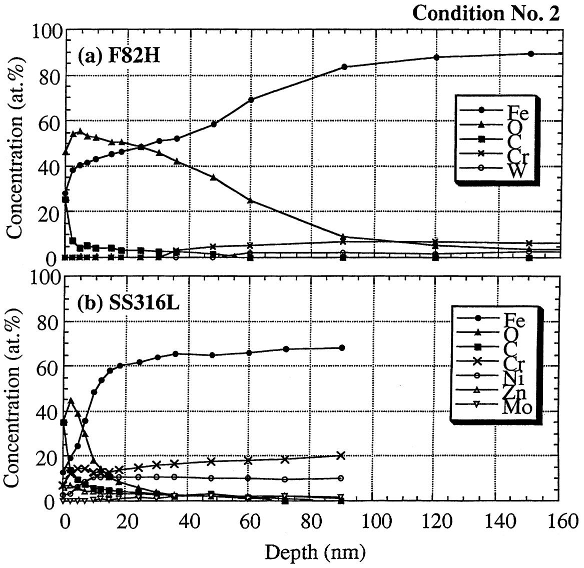


Fig. 3. The depth profiles of surface atomic composition of F82H and SS316 exposed to hydrogen atmosphere under the condition No. 2.

The results of the surface analysis for the specimens exposed to hydrogen atmosphere (test condition No. 2) are shown in Fig. 3 for both F82H and SS316L. Oxidation layer of F82H formed on F82H surface was found deeper than that of SS316L. The thickness of the surface oxidation layer was estimated to be increased up to 56 nm for F82H, while less increased up to 9 nm for SS316L. The thickness of the oxidation layer formed on F82H surface was six times as thick as SS316L, suggesting that oxygen is more diffusive in F82H than SS316L and F82H is more reactive than SS316L under the conditions examined. The results of No. 3 specimens are shown in Fig. 4. The thickness of the surface oxidation layer was further increased up to 84 nm for F82H and 55 nm for SS316L. These thickness of the oxidation layers are summarized in Table 2. It was found that the surface oxidation layer of F82H showed saturating tendency owing to chromium surface segregation in spite of the accelerated test conditions, while SS316L showed increasing oxidation layer together with the increase of the temperature and hydrogen pressure.

3.2. Quantitative analysis

The hydrogen effects on the mechanical properties of SS316L have been already studied [7] and it was reported that the absorbed hydrogen of 40 ppm causes a slight reduction in ductility of the steel. Similarly, we examined the hydrogen effect on the mechanical properties of F82H and 316L steel. A quantitative analysis of hydrogen contents by a gaschromatograph was performed in order to measure the absorbed hydrogen of F82H and 316L test specimens quantitatively. The

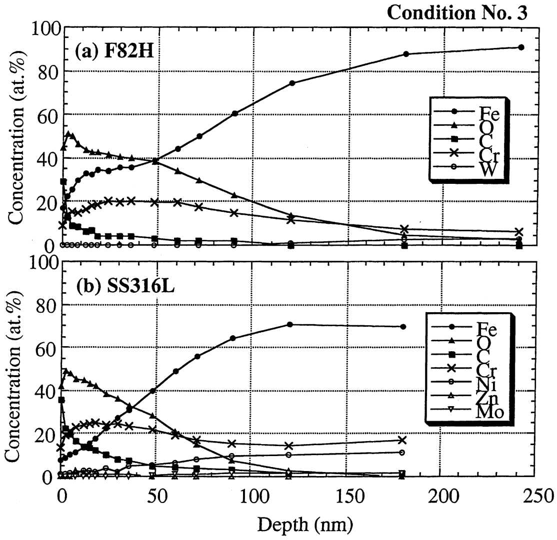


Fig. 4. The depth profiles of surface atomic composition of F82H and SS316 exposed to hydrogen atmosphere under the condition No. 3.

Table 2
Thickness of measured oxidation layers

No.	F82H (nm)	SS316L (nm)
1	7	7
2	56	9
3	84	55

Table 3
Hydrogen contents by quantitative analysis

No.	F82H (ppm)	SS316L (ppm)
1	<0.5	2.1
2	<0.5	1.8
3	<0.5	3.2

tensile specimens were used in this measurement after the exposure to hydrogen atmosphere under the conditions listed in Table 1. The results are summarized in Table 3. Hydrogen contents of F82H specimens are negligibly small below the detectable level (5 ppm), and it was found that F82H does not absorb any significant amount of hydrogen under the test conditions examined. On the contrary, hydrogen contents of SS316L specimens were detectable in the range of a few ppm, which was deduced to be coming from the as-received SS316L matrix rather than the absorbed ones.

3.3. Tensile tests

Tensile tests have been performed at room temperature for both of the F82H and SS316L test specimens exposed to the hydrogen atmosphere. Measured tensile strengths and total elongation of the F82H and SS316L test specimens are shown in Fig. 5. The horizontal axis shows the condition number of hydrogen exposure listed in Table 1. All of the measured tensile strength of the F82H specimens were close to or slightly over 68 kg/mm^2 and almost the same strength as the base materials reported in the literature [8]. Measured total elongation of the F82H specimens were in the range from 0.15 to 0.20, almost the same as those reported in the previous studies [9]. There are no clear dependence of tensile properties on these test conditions, and it can be concluded that tensile properties of F82H were not affected by exposing this steel under hydrogen atmosphere and elevated temperature examined. Similar tendency was also confirmed for SS316L.

3.4. Impact test

Charpy impact tests have been performed in order to evaluate the effect of hydrogen atmosphere on impact properties. The impact tests have been conducted at room temperature. Test results are given in Fig. 6. It can be seen that the impact energies of the F82H specimens exposed to hydrogen atmosphere under three different conditions stayed within the range between 16.8 and 18.4 kgf m/cm^2 and showed no clear differences among the three conditions examined. These results means that the impact property of F82H steel was not affected by exposing it to the hydrogen atmosphere within the range examined.

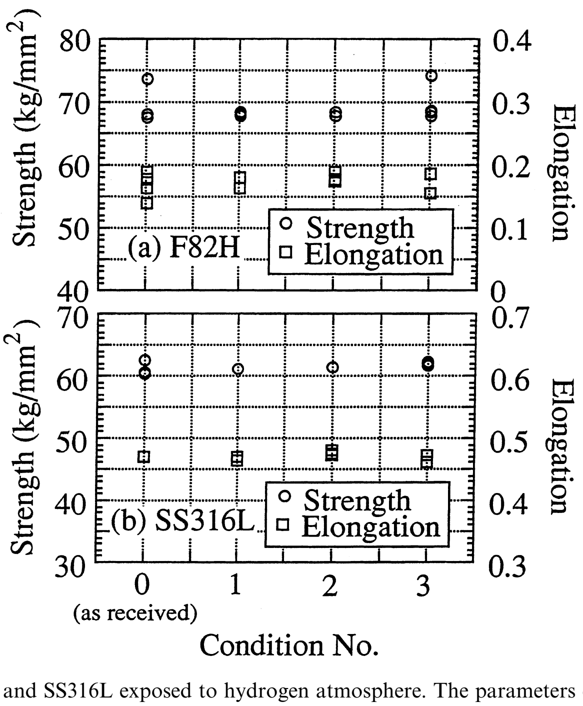


Fig. 5. Tensile properties of F82H and SS316L exposed to hydrogen atmosphere. The parameters of each condition No. are listed in Table 1.

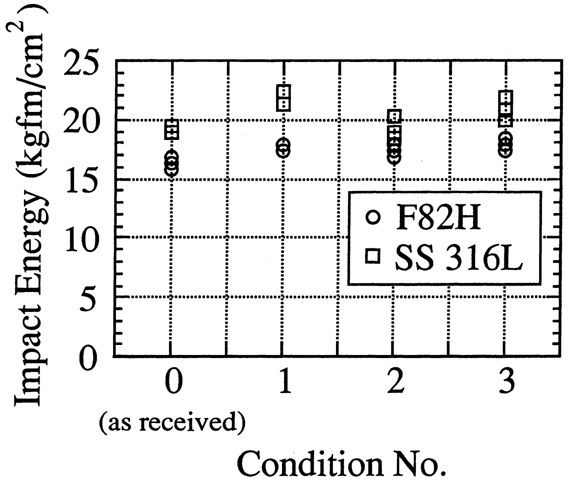


Fig. 6. Impact properties of F82H and SS316L exposed to hydrogen atmosphere. The parameters of each condition No. are listed in Table 1.

4. Conclusions

The effects of hydrogen atmosphere on mechanical properties and surface conditions of the reduced activation ferritic steel F82H have been studied. Test specimens were exposed to hydrogen gas atmosphere under three different conditions simulating the conditions anticipated in a fusion reactor. By using these specimens, surface analyses were performed by means of Auger electron spectroscopy in order to evaluate the depth profiles of surface atomic composition. Surface oxidation layer was observed with a depth of up to 56 nm, and the oxidation layer thickness of F82H was found six times larger than that of SS316L. However, saturating tendency was observed owing to chromium surface segregation in spite of the severe test conditions. A quantitative analysis of absorbed hydrogen was performed by a gaschromatograph, and the amount of absorbed hydrogen in F82H test specimens was found negligibly small below the detectable level of 5 ppm. Tensile tests and Charpy impact tests were carried out with the specimens exposed to hydrogen atmosphere, no degradation effects were observed on these mechanical properties within the parameter ranges examined. And it can be concluded mechanical properties of F82H steel is less sensitive to hydrogen atmosphere than SS316L, and no serious issues can be identified with these regards.

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References

[1] M. Tamura et al., J. Nucl. Mater. 155–157 (1988) 620–625.
[2] Y. Seki et al., 13th Conf. on Plasma Phys. and Cont. Nucl. Fusion Res., IAEA-CN-53/G-1-2 (1990).
[3] H. Takatsu et al., Presented at 4th Internat. Symp. on Fusion Nucl. Technol., Tokyo, Fusion Eng. Des. (1997) to appear.
[4] K. Shiba et al., Fusion Mater. Semi-annual Report, DOE/ER-0313/20 (1996) 190.
[5] K. Shiba, A. Hishinuma, A. Tohyama, K. Masamura, JAERI-Tech 97–38 (1997).
[6] K. Odaka et al., J. Plasma and Fusion Res. 73 (9) (1997) 1001.
[7] Y.Y. Li, Z.S. Xing, J. Nucl. Mater. 169 (1989) 151.
[8] M. Tamura, H. Hayakawa, M. Tanimura, A. Hishinuma, T. Kondo, J. Nucl. Mater. 141–143 (1988) 1067.
[9] N. Yamanouchi, M. Tamura, H. Hayakawa, A. Hishinuma, Kondo, J. Nucl. Mater. 191–194 (1992) 822.