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Correlation between hydrogen distribution in V–4Cr–4Ti alloy and impact strength

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ABSTRACT

Distribution of hydrogen isotopes was examined by tritium radioluminography using imaging plates for specimens of a V–4Cr–4Ti alloy prepared from two different plates fabricated with cold rolling under two different conditions (93% and 96% reduction in thickness) and recrystallization annealing. Influence of hydrogen on impact strength was also studied. Radioluminographs showed that tritium was concentrated in band-like regions where Ti(C,N,O) precipitates were densely distributed. The extent of tritium accumulation in the band-like regions and their shapes, however, were different between the two types of specimens; the band-like regions were thinner and more elongated, and the trapping effects of precipitates was smaller for the specimen prepared with higher extent of cold working. On the other hand, this type of specimen showed lower susceptibility to hydrogen embrittlement. These observations indicated that precipitate distribution prepared with higher extent of cold working is preferable to provide better durability against hydrogen embrittlement.

1. Introduction

Hydrogen embrittlement of structural materials is important issue for safety aspect of fusion reactors. From this view point, influence of hydrogen on mechanical properties of V alloys has been studied extensively [1–7]. Loomis et al. [1] have examined the effects of hydrogen on impact energy of various V alloys including a V–4Cr–4Ti alloy currently recognized as a reference material. They observed significant reduction in absorbed energy at hydrogen concentration of 1200 appm for a V–4Cr–4Ti alloy in a wide temperature range from −200 to 20 °C [1]. Results of tensile tests on V–4Cr–4Ti alloys carried out by several different research groups showed that the severe embrittlement starts at hydrogen concentration of few at.%, depending on various factors such as heat treatment conditions and oxygen concentration [2–7]. In these studies, correlations between average hydrogen concentration and mechanical properties were examined. The specimens fabricated under different conditions, however, should have different microstructures, and consequently different distributions of hydrogen. Indeed, the present authors have found by tritium radioluminography that distribution of hydrogen in a V–4Cr–4Ti alloy is strongly affected by that of precipitates consisting of Ti, C, N and O [8,9]. Therefore, it is necessary to understand the correlation of microstructure, hydrogen distribution and susceptibility to hydrogen embrittlement.

In the present study, two types of specimens of a V–4Cr–4Ti alloy were prepared from plates fabricated with different reduction in thickness in cold working. Then the correlation of microstructure, hydrogen distribution and impact strength was examined.

2. Experimental

Specimens for tritium radioluminography and impact tests were prepared from two different cold-rolled plates of a V–4Cr–4Ti alloy (NIFS-HEAT-2 [10,11]). The thickness and the reduction of thickness by cold rolling were 4.0 mm and 96% for one plate, and those for another were 6.6 mm and 93%. The specimens prepared from the former plates are hereafter denoted as 4.0t specimens, and those from the latter as 6.6t specimens. Sheet type specimens (6.5 × 4.0 × 0.5 or 6.5 × 6.6 × 0.5 mm) were taken for radioluminography in the direction corresponding to LS view described in Ref. [10] to observe tritium distribution in cross sections. The miniature specimens for impact tests (1.5 × 1.5 × 20 mm) were cut from center of thickness of the plates along rolling direction; V-notch (0.3 mm depth) was prepared on the side parallel to normal direction. This orientation is equivalent to LT in Ref. [10]. All specimens were used under recrystallized conditions (1273 K, 2 h).

The surfaces of specimens for tritium radioluminography were polished with abrasive papers and mirror-finished by using alumina paste with 0.06 μm particles. Tritium diluted by deuterium ($T/D = 5.6 \times 10^{-3}$) was introduced into the specimen by gas

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absorption method using vacuum apparatus at 673 K in the manner described elsewhere [9]. The total concentration of hydrogen isotopes (D and T) was adjusted to be 100 or 1500 appm. After the tritium absorption, the specimen was kept at 673 K in vacuum for 30 min to obtain uniform distribution of tritium through the specimen thickness. Then, the specimen was placed on an imaging plate (IP), Fujifilm TR2025, for 24 h at room temperature. Two-dimensional mapping of β-ray dose was obtained by measuring the intensity of photo-stimulated luminescence (PSL) with an imaging analyzer (Fujifilm BAS-5000). The spatial resolution was $25 \times 25\ \mu m$.

Influence of hydrogen on impact energy was examined with two series of experiments. Firstly, hydrogen concentration in the specimens was adjusted to be 1500 appm, and temperature dependence of absorbed energy was measured in a range from 77 K to room temperature. Secondly, the specimen temperature was adjusted to be 173 K, and the hydrogen concentration was varied up to 6000 appm. Hydrogen was introduced by a gas absorption method in the similar manner to the tritium loading at temperatures of 423 K (1500–6000 appm) or 673 K (below 1500 appm). The specimen was kept in vacuum at these temperatures until the mean diffusion length of hydrogen became sufficiently larger than 1.5 mm.

3. Results and discussion

Typical examples of the radioluminographs at total concentration of D and T of 1500 appm are shown in Fig. 1. Change in color from blue to red indicates increase in the tritium concentration. Bands of tritium-accumulated regions are clearly observed along the rolling direction. It is known that these bands of tritium-accumulated regions correspond to the areas where precipitates consisting of Ti, C, N and O are densely distributed [8,9]. Namely, hydrogen isotopes are trapped in/around the precipitates. The band-like regions are thinner and more elongated in 4.0t specimen due to higher degree of cold working. Difference in the tritium concentration between the band-like regions and the alloy matrix is smaller in 4.0t specimen than in 6.6t specimen; the fraction of area with low tritium concentrations indicated by green is significantly smaller in the former. Results of line analyses given in Fig. 2 also show such tendency; the difference between the minimum and the maximum values of PSL intensity is much smaller for 4.0t specimen. These observations indicate that trapping effects by Ti(C,N,O) precipitates are weaker in 4.0t specimens. Such difference in the trapping effects can be ascribed to that the volume fraction of precipitates in 4.0t specimen is smaller than that in 6.6t specimen. It is known that the precipitates are partly dissolved in the matrix during annealing at 1273 K [11]. One of the possible explanations of the difference is that higher degree of cold working led to the finer precipitates and consequently faster dissolution into the matrix during recrystallzation annealing. Similar difference in tritium distribution was also observed between 4.0t and 6.6t specimens at the total concentration of D and T of 100 appm.

Fig. 3(a) shows the temperature dependence of impact energy at hydrogen concentration of 1500 appm or without hydrogen charging. In the case of 4.0t specimen, no noticeable influence of hydrogen was observed at room temperature and 173 K. The impact energy started to decrease at 143 K and took the minimum value at 103 K. In the case of 6.6t specimen, significant reduction in impact energy was induced by hydrogen even at 173 K. Namely,

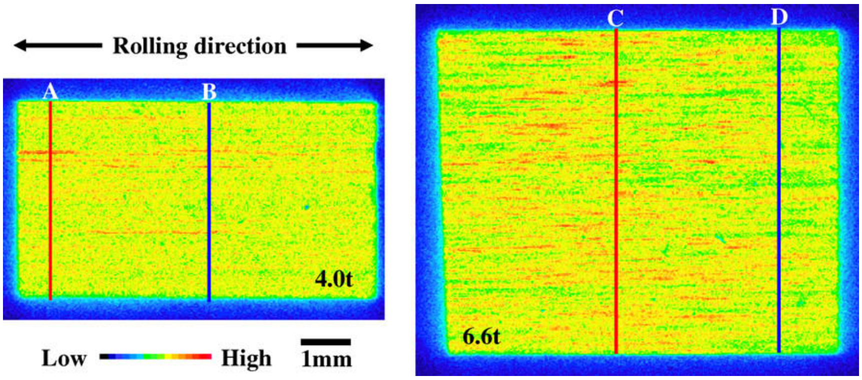

Fig. 1. Radioluminographs of 4.0t (left) and 6.6t (right) specimens at total concentration of D and T of 1500 appm. Lines indicate the positions of line analyses shown in Fig. 2.

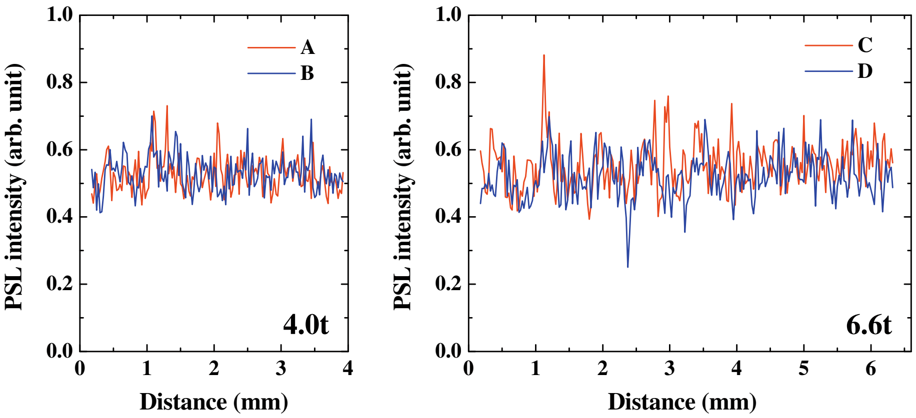

Fig. 2. Line profiles of PSL intensities for 4.0t (left) and 6.6t (right) specimens at the positions indicated in Fig. 1.

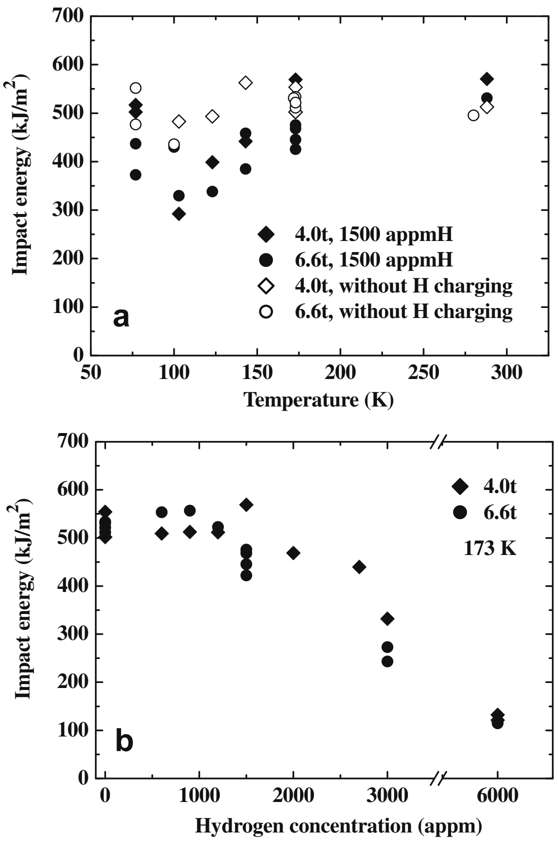

Fig. 3. Dependence of impact strength on temperature (a) and hydrogen concentration (b).

influence of hydrogen was observed at higher temperature for 6.6t specimen than for 4.0t specimen. The dependence of impact energy on hydrogen concentration at 173 K is shown in Fig. 3(b). The impact energy of 6.6t specimen started to drop at 1500 appm, while no significant degradation was observed for 4.0t specimen at this hydrogen concentration; the impact energy obtained for 4.0t specimen at 2700 appm was comparable with that for 6.6t specimen at 1500 appm.

The above-mentioned observations clearly indicate that susceptibility of 4.0t specimen to hydrogen embrittlement was lower than that of 6.6t specimen under the present conditions. One of the possible explanations is severer effects of local embrittlement in the band-like regions for 6.6t specimens. In 4.0t specimens, the extent of tritium accumulation in the band-like regions was smaller. In addition, the thinner and more elongated shape along the perpendicular direction to the propagation direction of primary crack is preferable to mitigate the effects of local embrittlement. Detailed observations of fracture surfaces are necessary for better understanding of underlying mechanism. Nagasaka et al. [10] examined the impact strength of NIFS-HEAT-2 specimens with different degree of cold working, i.e., from 74% to 98%, at 77 K without hydrogen charging, and reported that the specimens with higher degree of cold working had higher impact energy. Such correlation between extent of cold working and impact strength held also for hydrogenated specimens; the present results do not indicate the necessity of modification in optimization strategy for fabrication conditions. The influence of hydrogen on impact strength observed in the present study was much weaker than that reported by Loomis et al. (Fig. 1(d) in Ref. [1]). This difference can be attributed to smaller specimen size, higher degree of cold working, and lower concentration of oxygen in the present specimens; it is known that oxygen enhances hydrogen embrittlement of this type of alloys [2–4].

4. Conclusions

(1) Hydrogen isotopes were concentrated in band-like regions where Ti(C,N,O) precipitates were densely distributed. Such band-like regions were thinner and more elongated in 4.0t specimen than 6.6t specimen. The trapping effects by precipitates for hydrogen isotopes were smaller in the former specimen than in the latter.
(2) The impact tests showed that the susceptibility of 4.0t specimen to hydrogen embrittlement was lower than that of 6.6t specimen.
(3) The present results showed that precipitate distribution prepared with higher degree of cold working is preferable to provide higher impact energy for hydrogenated specimens as previously observed for non-hydrogenated specimens [10].

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