Effects of Inclusions on Fracture Toughness of Reduced-Activation Ferritic/Martensitic F82H-IEA Steels

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Reduced activation ferritic/martensitic steels (RAFs) are recognized as the primary candidate structural materials for fusion blanket systems. F82H is the RAF which has been developed and studied in Japan, and F82H-IEA heat, one of the world’s first 5 ton heats of RAF, was provided and evaluated in various countries as a part of the IEA collaboration on fusion materials development. A problem with the steel is that some fracture toughness values obtained by 1T type of compact tension (1TCT) specimens showed very low values in ductile brittle transition region. There might be several reasons for the scatter, and one of them could be related to the microstructural inhomogeneity of F82H-IEA. In the present study, this possibility was investigated focusing on inclusions formed in a plate of F82H-IEA steel by scanning (SEM) and transmission electron microscopy (TEM) equipped with EDS.

The plates examined in this study were obtained from F82H-IEA heat no. 9753, nominally Fe-7.5Cr-2W-0.15V-0.02Ta-0.1C, in mass%. Analyses by SEM and TEM for the plates revealed that Ta does not form MX precipitates, but instead, it forms composite Al₂O₃-Ta(V,Ti)O oxide, or single phase Ta(V)O oxide. The composite inclusions are rather dominant in the plate obtained from the bottom of the ingot, but not in the plate from the middle of the ingot. SEM observations also revealed that composite oxide tended to be observed at the crack-initiation site. These results suggest that the scatter of toughness values may be correlated with this microstructural inhomogeneity.

Keywords: reduced-activation ferritic/martensitic steels, neutron irradiated, inclusion, Ta oxide, toughness, master curve

1. Introduction

In the current Japan Atomic Energy Agency (JAEA; formerly JAERI) – U.S. Dept. of Energy (DOE) fusion materials collaboration program, the ductile-brittle transition temperature (DBTT) of the reduced-activation ferritic/martensitic steels (RAFs) is to be determined by the master curve (MC) method,¹,² in which the DBTT of ferritic steels is characterized in terms of a fracture toughness reference temperature, Tₒ, as defined in the ASTM standard E1921. This method works when the transition fracture toughness values follow the MC, and once the value is scaled properly, the MC is usually independent of the type of steel or the type of test specimen. The problem with F82H-IEA steel, one of the RAFs being examined in this collaboration, is that some fracture toughness values obtained by 1T type of compact tension (1TCT) specimens from two plates showed very low values that fell outside the tolerance bands of the MC in the ductile/brittle transition temperature range (Fig. 1),³ although the toughness values obtained from the other plates did not show such distribution.⁴ There may be several reasons for the scatter, and one of them could be the microstructural inhomogeneity of F82H-IEA. In this study, this possibility was investigated in terms of the types and distribution of inclusions.

2. Experimental

Plates examined in this study were obtained from F82H-IEA heat no. 9753, nominally Fe-7.5Cr-2W-0.15V-0.02Ta-0.1C, in mass%. The microstructures of plate numbers 2W-4 (15 mm thick plate) and 31W-3 (25 mm thick plate), obtained from the middle and the bottom-end section of the ingot, respectively, were examined. Chemical compositions and heat treatment conditions of these materials alloys are shown in Table 1.

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Table 1 Chemical composition (in mass% for C, Cr, W, V, Ta, and in mass ppm for Ti, N, O, Al, S), and heat treatment conditions of F82H-IEA heat.

<table>
<thead>
<tr>
<th></th>
<th>C</th>
<th>Cr</th>
<th>W</th>
<th>V</th>
<th>Ta</th>
<th>Ti</th>
<th>N</th>
<th>O</th>
<th>Al</th>
<th>S</th>
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<tbody>
<tr>
<td>F82H-IEA</td>
<td>0.1</td>
<td>7.7</td>
<td>2.00</td>
<td>0.15</td>
<td>0.02</td>
<td>30</td>
<td>65</td>
<td>74</td>
<td>10</td>
<td>8</td>
</tr>
</tbody>
</table>

Heat treatment condition: 1313 K × 40 min + 1023 K × 1 h

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Scanning Electron Microscope (SEM) observations were performed on specimens that were etched by the Selective Potentiostatic Etching by Electrolytic Dissolution (SPEED) method, in which all precipitates, inclusions and metastable phases remain unetched. SEM observations on the fractured specimens with toughness values outside the tolerance bands of the MC were performed. SEM observations were also performed on the fractured surfaces of fracture-toughness-tested specimens, which data are shown in Fig. 1, and the specimens were taken from plate numbers 31W-3 and 42W-1 (25 mm thick plate) of F82H-IEA heat no 9753, obtained from the bottom of the ingot. The 0.4T CT specimens were taken from a tested 1T CT specimen which showed low energy fracture in the transition temperature region and tested to confirm the 1T CT results. The detail of fracture toughness tests are shown in another paper.3)

Transmission Electron Microscopy (TEM) and energy filtered TEM observations were performed with JEOL 2200FS and JEOL 2010FX at MUSTER facility of Institute

Fig. 2 SEM images for toughness-tested fracture surfaces of each test condition: (a) 1TCT from plate no. 42W-1 fractured at 88.6 MPa/m tested at 253 K, (b) 1T CT from plate no. 31W-3 fractured at 87.3 MPa/m tested at 233 K, (c) 0.4T CT from plate no.31W-3 fractured at 116 MPa/m tested at 223 K.

Fig. 3 SEM images for toughness-tested fracture surfaces and EDX spectra obtained from the oxides observed on the fracture-initiation positions. All specimens were obtained from plate no. 42W-1 tested at 173 K and fractured at (a) 71.5 MPa/m, (b) 75.2 MPa/m, and (c) 72.7 MPa/m.

Fig. 4 SEM images of (a) a single phase inclusion Ta(V)Ox, and (b) a composite inclusion, Al2O3-Ta(V,Ti)Ox. Bright contrasts correspond to Ta-rich regions, and dark contrasts correspond to Al-rich region, and typical EDX spectra obtained from these regions.
of Advanced Energy of Kyoto University. A cross sectional TEM thin film specimen of an inclusion was made utilizing focused ion beam (FIB) micro-processor with micro-sampling system, HITACHI FB-2000A at the WASTEF facility of JAEA. The details of the FIB process were given elsewhere.5)

3. Results and Discussion

Figure 2 shows the fracture surfaces of fracture-toughness-tested specimens of various sizes. The surfaces of all specimens shown here have inclusions at its fracture starting point. EDX analyses revealed that all of these are Ta- and Al-rich oxides and some of them show the presence of sulfur (Fig. 3).

SEM observations were performed on etched F82H plates obtained from different portions of the ingot, and the precipitates and inclusions were analyzed. It turned out that Ta does not form MX precipitates, but instead, it forms two types of oxide, one is composite oxides which are a mixture of $\text{Al}_2\text{O}_3$ and $\text{Ta}(\text{V},\text{Ti})\text{O}_x$ [$\text{Al}_2\text{O}_3\cdot\text{Ta}(\text{V},\text{Ti})\text{O}_x$], and the other is single phase oxides which are composed of $\text{Ta}(\text{Ti},\text{V})\text{O}_x$ only (Fig. 4). The size distribution of these inclusions was analyzed on the region 1 mm from the plate surface and $t/2$ section of the plate obtained from the middle of the ingot (no. 2W-4), and from the bottom of the ingot (no. 31W-3) (Fig. 5). The results revealed that number density of oxides are high in the region near the surface, regardless to the portion of the ingot from which the plate obtained. In the $t/2$ section of the plate, the composite oxides are rather dominant in the plate obtained from the bottom of the ingot, but not in the plate from the middle of the ingot. The single phase oxide is dominant in the $t/2$ section of the plate from the middle of the ingot. It was also revealed that those composite inclusions found on the prior austenitic grain boundaries tend to have Mn- and S-rich compounds on its surface (Fig. 6). The Mn- and S-rich compounds are tend to found on the $\text{Ta}(\text{V},\text{Ti})\text{O}_x$ part of composite inclusions. EDS mapping and TEM images suggests the presence of Mn- and S-rich compound on the intersection of boundary and $\text{Ta}(\text{V},\text{Ti})\text{O}_x$ part of composite inclusions, and the compound do not include any other elements (Fig. 7).

These results suggest that the scatter of toughness values may be correlated with this inhomogeneous distribution of composite inclusions, as the scattered toughness data shown in Fig. 1 were acquired from the plate obtained from the bottom of the ingot at which the composite inclusions are dominant at $t/2$ section of the plate. It should be noted that there is the possibility that the Mn- and S-rich compounds observed on composite inclusion may play a key role for the low toughness in the transition temperature region, and also it should be noted that the sulfur contents in these plates are very low, about 8 ppm.

4. Summary

Microstructural inhomogeneity was investigated on F82H-IEA heat no. 9753, in which the toughness shows unexpected low energy fracture in the transition temperature region. The following conclusions may be drawn:
Inclusions were found on almost all fracture initiation points of the specimens which were tested in the transition temperature region and fractured at low energy.

Two types of inclusions were found in F82H plate; a composite oxide inclusion composed of Al$_2$O$_3$ and Ta(V,Ti)O, and a single phase Ta(Ti,V)O oxide.

The composite inclusions were dominant in the middle and surface section of those plates taken from the bottom of the ingot, but not in the plate taken from the middle of the ingot.

The single phase inclusions are dominant in the middle section of the plate from the middle of the ingot.

Mn- and S-rich compounds were observed on the Mn- and S-rich compounds were observed on the Ta(V,Ti)O part of the composite inclusions located on prior austenitic grain boundaries.

Mn- and S-rich compounds were also observed on the composite oxides found at the crack initiation points of the low energy fractured specimens.

REFERENCES