Study on the Segregation Behavior in SA508 Gr. 4N Low Alloy Steel with Mn Contents Variation

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1. Introduction

It is generally known that SA508 Gr.4N low alloy steel has an improved fracture toughness and strength, compared to commercial low alloy steels such as SA508 Gr.3 and SA533B which have lower than 1% Ni. Higher strength and fracture toughness of low alloy steels could be achieved by adding the Ni and Cr. So there are several researches on SA508 Gr.4N low alloy steel for a RPV application[1]. The operation temperature of a reactor pressure vessel is more than 300 ℃ and it operates for over 40 years. Therefore, in order to apply the SA508 Gr.4N low alloy steel for a reactor pressure vessel, it requires a phase stability in the high temperature range including temper embrittlement resistance. Although no brittlement has been reported in SA508 Gr.4N low alloy steel, we need to evaluate the temper embrittlement phenomena on SA508 Gr.4N for an RPV application. In a previous study, we have concluded that additional Mn may accelerate the temper embrittlement effect in SA508 Gr.4N low alloy steel[2]. So we need to examine the reason why Mn changes the susceptibility to temper embrittlement in SA508 Gr.4N.

In this study, we have performed a Charpy impact test of SA508 Gr.4N low alloy steel at varing Mn contents. The mechanical properties of these low alloy steels after a long-term heat treatment(450°C, 2000hr) are evaluated. Then, the images of the fracture surfaces are observed and a grain boundary segregation is analyzed by AES and SIMS. We also analyze the grain boundary structures of the low alloy steels with EBSD.

2. Experimental Procedure

Three types of pressure vessel steels with different Mn contents were selected for this study. The chemical compositions of the steels are given in Table 1. A model alloy KL4-Ref with a typical composition of the SA508 Gr. 4N steel was arranged as a reference alloy within ASME specified composition. It was planned to study the temper embrittlement effect in the SA508 Gr. 4N low alloys steel by changing Mn contents(KL4-Mn1, KL4-Mn2). Model alloys were austenitized at 880 °C for 2 hours followed by an air cooling, and then tempered at 660 °C for 10 hours. After the tempering process, model alloys were treated at 450 °C for 2000 hours, which can reveal the temper embrittlement phenomena efficiently[3].

Impact transition curves were obtained using standard Charpy V-notched specimens and using SATEC-S1 impact test machine with maximum capacity of 406J in a temperature range of -196℃ to 150℃. The index temperatures were determined from fitted Charpy curves as the temperature corresponding to the Charpy energy values of 48J and 68J.

The observations of the fractures were conducted using scanning electron microscope (SEM). The specimens were examined using SEM-6300 scanning electron microscope. Auger electron spectroscopy was used to monitor grain boundary segregation in the model alloy. All samples were fractured at low temperature (lower than -150 °C) in $2x10^{-10}$ torr, and the fracture surfaces were analyzed at 5kV. A ULVAC PHI 700 auger electron microscope was employed for the analysis.

Grain boundary segregation behavior is evaluated by Secondary Ion Mass Spectroscopy (SIMS). The specimens were prepared in a disk 1mm in diameter and 2mm in thickness. They were analyzed by IMS-6f secondary ion mass spectroscopy. Grain boundary structures were observed by Electron Back-Scattered Diffraction (EBSD) using JSM-700F field-emission scanning electron microscope.

Table 1. Chemical compositions of steels. (wt%)

	C	Mn	Ni	Cr	P	Fe
KL4-Ref	.190	.297	3.59	1.79	.002	Bal.
KL4-Mn1	.211	.111	3.64	1.85	.002	Bal.
KL4-Mn2	.210	.515	3.63	1.86	.002	Bal.

3. Experimental Results and Discussion

Fig. 1 shows the Charpy impact test results. It is shown that the transition curves of the KL4-Mn2 and KL4-Ref are shifted to a higher temperature region. From the transition curve, it is apparent that both of the higher Mn steels KL4-Ref and KL4-Mn2 experienced a greater upward shift in the index transition temperature(T_{41J}) after long term heat treatment. It gives the initial T_{41J} of -127.8°C in KL4-Ref and -130.4°C in KL4-Mn2, compared with -89.5°C and -54.5°C after ageing, respectively. On the other hand, the index transition temperature was slightly increased from -123.5°C to -113.6°C in the KL4-Mn1.

In order to analyze the fracture behavior, the fracture surfaces of the model alloys are observed by SEM. Fig. 2 shows the fracture surface of the KL4-Mn1 and KL4-

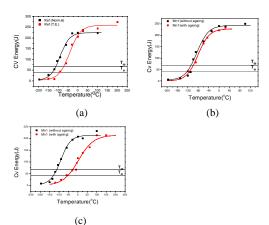


Fig. 1 Charpy transition curves of the (a) KL4-Ref, (b) KL4-Mn1 and (c) KL4-Mn2

Mn2 in the lower transition region. In the SEM observation results, the fracture behavior of the KL4-Mn2 is changed from a partial intergranular to an almost intergranular manner after a long-term heat treatment. However, the fracture appearance of the KL4-Mn1 does not show any intergranular behavior in either condition. Based on the mechanical test and fracture surface analysis results, KL4-Mn2 (0.515wt% Mn) is the most severely embrittled after the long-term heat treatment while KL4-Mn1 (0.111wt% Mn) shows little embrittlement behavior in spite of same P contents. Therefore, it is considered that the susceptibility to temper embrittlement in SA508 Gr.4N low alloy steel is reduced with decreasing Mn contents.

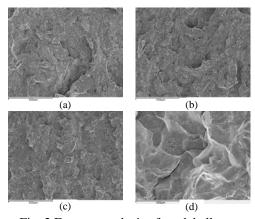


Fig. 2 Fracture analysis of model alloys Before ageing (a)KL4-Mn1 (b)KL4-Mn2, After ageing (c) KL4-Mn1, (d) KL4-Mn2

It is generally known that the cause of temper embrittlement is a grain boundary segregation of the impurity elements such as P and Sb[4]. In the research, we also observed the segregation of P in embrtteled model alloy by using AES[2]. Therefore, we can suppose that the Mn affect the impurity segregation level in the model alloys and it causes different embrittlement behavior with different Mn contents. In order to investigate the Mn effect in the model alloys,

we calculated the diffiusivity of P with changing Mn contents using DICTRA(Fig.3), which can calculate the kinetic behavior based on thermodynamics. In the calculation result, the diffusivity of the P is increased with increasing Mn content. So the additional Mn accelerates the grain boundary segregation of P, which causes the temper embrittlement.

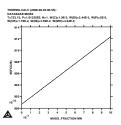


Fig. 3 Calculation results of the mobility of P with Mn contents

In addition to segregation enhancement, increased Mn enhances the hardenability of the steel and it may affect the change of microstructure including grain size reduction. The embrittlement behavior became more severe as the grain size decreased. And also, It is reported that Mn segregated itself in the grain boundary[5] and it contributes to the embrittlement behavior[6]. The segregation of Mn and its microstructure changes will be dicussed with SIMS and EBSD in detail.

4. Summary

In this study, evaluation of the temper embrittlement on SA508 Gr.4N low alloy steel by a mechanical test and a fracture analysis was carried out. The most severe temper embrittlement occurred in KL4-Mn2, which has the highest Mn content. The reason of the temper embrittlement is a grain boundary segregation of the impurity element P. The differences in embrittlement behavior with different Mn model alloys are mainly caused by promotion of P segregation.

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