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Effects of the Microstructure on Segregation behavior of Ni-Cr-Mo High Strength Low Alloy RPV Steel

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1. Introduction

SA508 Gr.4N Ni-Cr-Mo low alloy steel has an improved fracture toughness and strength, compared to commercial Mn-Mo-Ni low alloy RPV steel SA508 Gr.3. Higher strength and fracture toughness of low alloy steels could be achieved by adding Ni and Cr. So there are several researches on SA508 Gr.4N low alloy steel for a RPV application[1]. The operation temperature and time of a reactor pressure vessel is more than 300℃ and over 40 years. Therefore, in order to apply the SA508 Gr.4N low alloy steel for a reactor pressure vessel, it requires a resistance of thermal embrittlement in the high temperature range including temper embrittlement resistance. S. Raoul [2] reported that the susceptibility to temper embrittlement was increasing a function of the cooling rate in SA533 steel, which suggests the martensitic microstructures resulting from increased cooling rates are more susceptible to temper embrittlement. However, this result has not been proved yet. So the comparison of temper embrittlement behavior was made between martensitic microstructure and bainitic microstructure with a viewpoint of boundary features in SA508 Gr.4N, which have mixture of tempered bainite/martensite.

In this study, we have compared temper embrittlement behaviors of SA508 Gr.4N low alloy steel with changing volume fraction of martensite. The mechanical properties of these low alloy steels) were evaluated after a long-term heat treatment(450℃, 2000hr. Then, the images of the segregated boundaries were observed and segregation behavior was analyzed by AES. In order to compare the misorientation distributions of model alloys, grain boundary structures were measured with EBSD.

2. Experimental Procedure

A model alloy of SA508 Gr.4N low alloy steel was selected for this study. The chemical compositions of the steels are given in Table 1. The model alloy of KL4 with a typical composition of the SA508 Gr.4N steel was arranged as a reference alloy within ASME specified composition. Model alloy was austenitized at 880℃ for 2 hours followed by different cooling rates (16℃/s, 0.47℃/s, and 0.05℃/s), and then tempered at 660℃ for 10 hours. Each model alloys were named on the dependence of cooling rate. 16℃/s is named as WQ, 0.47℃/s is named as AC, and 0.05℃/s is referred to FC. After the tempering process, the model alloys were treated at 450℃ 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 an 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 a scanning electron microscope (SEM). The specimens were examined using an SEM-6300 scanning electron microscope. Auger electron spectroscopy was used to monitor the grain boundary segregation in the model alloy. All samples were fractured at low temperature (lower than -150℃) in $2x10^{-10}$ torr, and the fracture surfaces were analyzed at 5kV. A ULVAC PHI 700 auger electron microscope was employed for the analysis.

The grain boundary segregation behavior was observed by selective boundary etching method. The specimens were etched in an aqueous saturated picric acid with a 1g of wetting agent (sodiumtridecyl-bezenesulfonate) at 25℃ for 9ks. The grain boundary structures were observed by Electron Back-Scattered Diffraction (EBSD) using a JSM-700F field-emission scanning electron microscope.

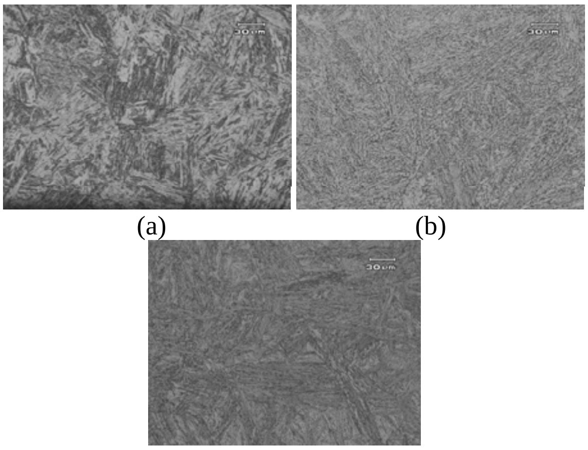
Table 1. Chemical compositions of steels. (wt%)

	C	Mn	Ni	Cr	P	Fe
KL4	0.20	0.30	3.64	1.80	.029	Bal.

3. Experimental Results and Discussion

Fig. 1 shows the optical micrographs of the as quenched model alloys. The cooling rate of 16℃/s (WQ) reveals the almost martensitic microstructure, while the lower bainitic microstructure is observed in the cooling rate of 0.05℃/s (FC). The model alloy of AC shows a mixed microstructure of martensite/bainite, and it has been reported that the volume fraction of martensite in AC is about 70%.

Fig. 2 shows the Charpy impact test results. From the transition curve, it is shown that the impact toughness of FC is slightly lower than other model alloys before ageing. However, WQ experienced a greater upward shift than FC in the index transition temperature(T_{68J}) after long term heat treatment. It gives the T_{68J} of 117.0℃, 96.7℃ and 80.6℃ after ageing in WQ, AC



(a) (b)

(c)

Fig. 1 Optical micrographs of the as-quenched (a) WQ, (b) AC and (c) FC

and FC, compared with -54.0℃, -56.2℃ and -29.7℃ in normal condition, respectively. Compare the value of transition temperature shift (TTS) with the volume fraction of martensite, TTS was linearly increased as the martensite fraction increases in spite of the same P contents. Thus it seems that the resistance of temper embrittlement is lower in martensitic microstructure than that of bainitic microstricture.

It is generally known that the cause of temper embrittlement is a grain boundary segregation of the impurity elements such as P and Sb. In addition, the segregation is thermodynamically favored to occur at high angle boundaries, while the boundaries it does not segregated in low energy boundaries such as low angle or coincidence site lattice(CSL) boundaries [4]. In the previous work, it has been concluded that there was a larger portion of high energy boundaries inside the prior austenite grains in the tempered bainitic structure than those of the tempered martensitic structure [5]. In order to compare the segregation behaviors between WQ and FC, the selective etching methods were employed. Fig. 3 shows the optical micrographs of the WQ and FC after selective etching. In the case of WQ, which has tempered martensitic structure, the prior austenite grain boundaries were significantly attacked by selective etchant, while the boundaries inside the prior austenite grains were almost not attacked. In contrast, the some of the boundary in FC which placed inside the prior austenite grain was slightly attacked by selective etchant.

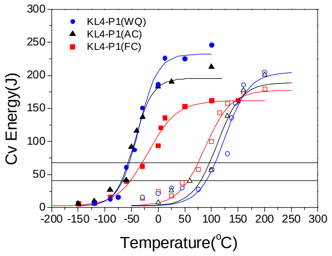
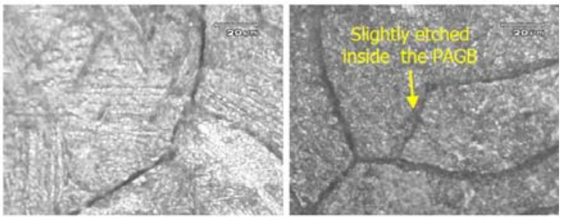


Fig. 2 Charpy transition curves of the model alloys with different cooling rates



(a) (b)

Fig. 3 Optical micrographs of a selectively etched (a) WQ and (b) FC

Thus the segregation behavior of P in the prior austenite grain boundaries would be much more reduced in FC than WQ after ageing, because the large amounts of segregation occurred in high energy boundaries inside the prior austenite grains. The differences between the tempered martensite and tempered bainite in the viewpoint of segregation behavior of P in the boundaries and the properties of boundaries will be dicussed with AES and EBSD in detail.

4. Summary

In this study, comparison of the temper embrittlement behaviors on tempered martensitic and tempered bainitic SA508 Gr.4N low alloy steel by a mechanical test and a microstructural analysis was carried out. The resistance of temper embrittlement was reduced as the volume fraction of martensite was increased. The differences in temper embrittlement behavior between tempered martensitic and tempered bainitic alloys are mainly caused by different portion of high energy boundaries inside the prior austenite grains.

Acknowledgement

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