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Effects of heat treatment conditions on high-temperature tensile properties in HT9 and Gr.92 steel

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1. Introduction

A Sodium-cooled Fast Reactor (SFR) is a reactor operated by high-energy neutrons that enables it to recycle the spent fuel from a conventional light water reactor. The fuel cladding tube is the most important safety barrier in fission nuclear reactors. It was occurred thermal creep and void swelling by fission gas at high temperature for service time. Ferritic-martensitic steels are being considered as an attractive candidate material for a fuel cladding of a SFR due to their low expansion coefficients, high thermal coductivities and excellent irradiation resistances to a void swelling [1]. HT9 steel (12CrMoVW) is initially developed as a material for power plants in Europe in the 1960. This steel has experienced to expose up to 200dpa in FFTE and EBR-II. Ferritic-Martensitic steel′s maximum creep strength in existence is 180Mpa for 106 hour 600℃, but HT9 steel is 60Mpa [2]. Because SFR is difficult to secure in developing and applying materials, HT9 steel has accumulated validated data and is suitable for SFR component. And also, because of its superior dimensional stability against fast neutron irradiation, Ferritic-martensitic steel of 9Cr and 12Cr steels, such as HT9 and Gr.92 are preferable to utilize in the fuel cladding of an SFR in KAERI. The objective of this study is to compare the effect of the heat treatment process on the mechanical properties of an HT9 and Gr.92 steel in the viewpoint of microstructure. Both HT9 and Gr.92 steel were normalized and tempered with various temperature settings and the vickers hardness test and tensile test were carried out to find the optimized heat treatment range. The microstructures observations were conducted using OM, and the grain boundary structures were observed by Electron Back-Scattered Diffraction (EBSD). Based on the mechanical properties obtained from the experimental studies, as well as from correlations with detailed information on boundary characteristics, the heat treatment effects between HT9 and Gr.92 steel are discussed.

2. Experimental Procedure

KAERI has manufactured a ferritic-martensitic cladding tube (12Cr1MoWV, HT9) in cooperation with a domestic steelmaking company. After making a steel ingot, followed by mutilple processes of tube reduction and subsequent heat treatment, a seamless HT9 tube with a 7.4 mm outer diameter, 0.56 mm thickness and 3000 mm length was manufactured. In addition, Gr.92 steel wire was also manufactured. To find the optimized heat treatment conditions of the HT9 and Gr.92 steel, it was normalized in a range of 950°C to 1100°C for 30 minutes followed by air cooling, and tempered in the range of 700℃ to 800℃ for 1 hour to change the microstructures. A tension test was carried out in accordance with the ASTM E8 specification. The strain rate was 0.005/min, and tests were performed from room temperature to 650℃.

Microstructure observations were conducted using optical microscopes and transmission electron microscopy (TEM) and image analyzer. Specimens for these observation were prepared by a grinding and polishing up to 0.25 ㎛ power, followed by etching using an etchant of 95 ml water + 3 ml nitric acid + 2 ml fluoric acid. To investigate the overall distribution of the carbides and to analyze the individual carbide particles in detail, carbon extraction replicas technique was employed. The etched specimens were then carbon coated, and the replicas were released by an electrochemical method with 1.6V in a solution of 90 ml methanol + 10 ml Hydrochloric acid. Thin-foils and carbon replicas were examined using JEOL JEM-2100FX

Mechanical properties were evaluated using vickers microhardness test (HM-122) and tensil test (INSTRON-3367). Vickers Microhardness test was carried with load of 500g.

The grain size and misorientation distributions were observed by by Electron Back-Scattered Diffraction (EBSD) using a JSM-700F field-emission scanning electron microscope.

3. Experimental Results and Discussion

Fig. 1 shows the optical micrographs of the Gr.92 steel normalized at 950℃ and 1100℃. The microstructure of the Gr.92 showed typical tempered martensite structure. As the temperature of the normalizing increases, the sizes of the prior austenite increase. Similar tendency was also observed in HT9 steel. The size of the prior austenite grains of 1000℃, 1050℃ and 1100℃ normalizing conditions were measured as 23.7 ㎛, 32.5 ㎛ and 40.9 ㎛, respectively. It is known that the A_{c1} temperature of HT9 steel is about 800℃, and thus the residence time at the austenite single phase will be longer with increasing normalizing temperature. This may be the reason for the growth of prior austenite grains normalized at higher temperature.

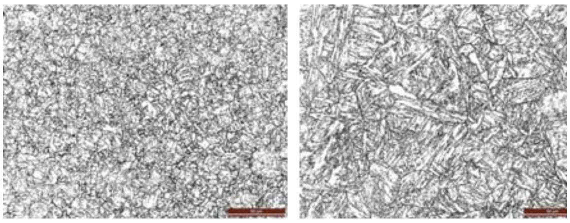
(a) (b)

Fig. 1 Optical micrographs of the Gr.92 steel normalized at (a) 950℃ and (b) 1100℃

Fig. 2 shows the Vickers hardness test results of Gr.92 steel with different heat treatment conditions. The hardness was linearly decreased with an increasing tempering temperature and it show higher values with an increasing normalizing temperature. Similar tendency were observed in yield strengths obtained from the room temperature test results. Both the yield stress and ultimate tensile stress decreased with an increasing tempering temperature. However, In the case of the samples normalized at 950℃, the strengths tested at 650℃ show linear behavior in spite of increasing tempering temperature. This phenomenon was also observed in HT9 steel. Both the yield stress and ultimate tensile stress decreased when the tempering temperature increased, while the ductility gradually increased. From this study, tempering at higher temperature increases the room-temperature cladding toughness, while tempering at lower temperature increases the 650℃ cladding toughness.

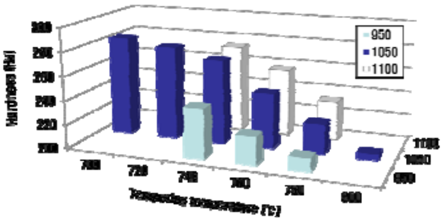

Fig. 2 Vickers hardness of the Gr.92 steel with different heat treatment conditions

To examine the packet sizes, we conducted the EBSD-measurements in the model alloys. Fig. 1(a) shows the analysis of the misorientation angles in model alloy. When analyzing as-quenched state, small peak is observed in the angle range of 27~33°, and the ∑11, ∑13, ∑39 boundaries were appeared. They were mainly observed in the austenite boundary and disappeared after tempering process. So these misorientation relationships must be formed in the austenite/martensite packet boundaries. It was also improved by the point-to-point misorientation profile between packet boundaries. Therefore, the average packet sizes on the EBSD map were measured at 15°. Both the HT9 and Gr.92 steel show significant decrease of effective grain size after 950℃ normalizing heat treatment. This could be explained the enhancement of high temperature strength in HT9 and Gr.92 steel, and these results will be quantitatively discussed with an EBSD analysis results in detail

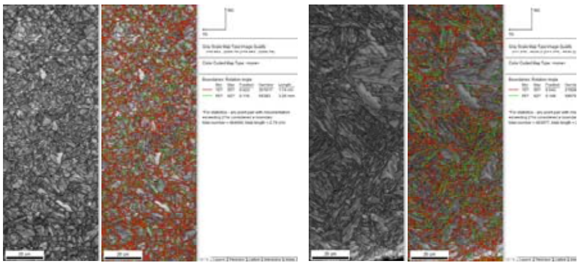
(b) (b)

Fig. Misorientation distributions of the Gr.92 steel normalized at (a) 950℃ and (b) 1100℃

4. Summary

In this study, comparison of the microstructure and mechanical properties on Gr.92 and HT9 steel with various heat treatment conditions were carried out. The size of prior austenite was decreased as the normalizing temperature was decreased. The differences in high temperature strength are mainly caused by different size of effective grains inside the prior austenite grains.

Acknowledgement

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