Effect of Heat treatment and Aging Conditions on the Microstructure and Mechanical Properties of HT9 Steel for Fuel Cladding Tube

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1. Introduction

The SFR (Sodium-cooled Fast Reactor) is a reactor that uses fast neutrons. It has attracted attention as an eco-friendly next-generation nuclear reactor, and has outstanding economics because of the recycling of the nuclear fuel used in the light water reactor, as well as the thermal and high neutron efficiencies [1].

The fuel cladding tube is the most important safety barrier in a fission nuclear reactor. Thermal creep and void swelling occur by fission gas at high temperature during service time. Ferritic-martensitic steels are being considered attractive candidate materials for a fuel cladding of the SFR owing to their low expansion coefficients, high thermal conductivities and excellent irradiation resistance to void swelling [2]. However, HT9 steel has a problem of a relatively low high-temperature strength and low creep strength. To solve this problem, a study was conducted to increase the high temperature strength by changing the intermediate heat treatment step in the fabrication process of ferritic-martensitic steel and controlling the microstructure and precipitate within the material [3]. Ferritic-martensitic steel was studied by varying the normalizing and tempering temperature to evaluate the mechanical properties [4]. The effects of the heat treatment and aging conditions on the microstructure and mechanical properties of HT9 steel were discussed. Each condition was achieved by substituting the tempering parameter and the relationship between the tempering parameter, and the mechanical properties were investigated to derive an equation.

2. Experimental Procedure

An HT9 tube with a 7.4 mm outer diameter, and 0.56 mm thickness was manufactured. The chemical composition of HT9 is given in Table 1.

Table 1. Chemical composition of HT9. (wt%)

Element	C	Cr	Mo	W	Nb	V	Fe
HT9	0.18	11.99	1.00	0.54	0.008	0.30	Bal

The HT9 tube was heated in an argon atmosphere in a sealed quartz tube. The heat treatment conditions of HT9 were normalized at 1000 to 1100℃ for 30 minutes followed by air-cooling to room temperature. The tempering treatment of the normalized specimens was carried out at 700 to 780℃ in 1 hour followed by air-cooling to room temperature. It was aged at 660℃ for 500, 1000, 3000, and 7000hours.

Microstructure observations were conducted using optical microscopes, transmission electron microscopy (TEM), and an image analyzer. The specimens used for these observation were prepared by 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 lath size, structure, and overall distribution of the carbides, and to analyze the individual carbide particles in detail, a thin-foil and carbon extraction replica technique was employed. Thin-foil specimens for TEM were prepared in a twin-jet electrochemical polisher operated at a flow rate of 25 cc/s and an applied voltage 25V, with 900 ml methanol + 100 ml perchloric acid under temperature conditions at -35℃. 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. The tin-foils and carbon replicas were examined using JEOL JEM-2100FX.

The mechanical properties were evaluated using a Vickers microhardness test (HM-122) and tensile test (INSTRON-3367). The Vickers microhardness test was carried out with a load of 500g. The HT9 tube was cut into 150mm pieces with a gauge length of 52-55 mm for the tensile test. The tensile test was carried out at a strain rate of 0.005mm/mm· min at room temperature and 650℃

3. Experimental Results and Discussion

3.1 Heat treatment condition on the microstructure and mechanical property

The HT9 cladding tube has a typical Martensite structure, and the PAG size (prior austenite grain size) increased with an increase normalizing temperature. The microstructure of the normalized specimens consisted of lath Martensite with a high dislocation density due to austenite to Martensite transformation. The tempered specimens showed a tempered lath Martensite structure with a low dislocation density because of the dislocation recovery. The lath width is not greatly affected by the tempering temperature change. A precipitate, unlike a lath structure, varies depending on the tempering temperature. Fig. 1 shows the precipitates and PAG size, as well as the lath width according to the tempering parameter. The PAG size indicates a similar value by increasing the tempering parameter. The PAG size was not affected by the tempering parameter. It is known that PAG size varies with the normalizing temperature. The lath width showed similar values by increasing the tempering parameter. The lath width was not affected by the tempering parameter. However, the size of the precipitate was increased by increasing the tempering parameter. The precipitate behavior was affected by the tempering parameter.

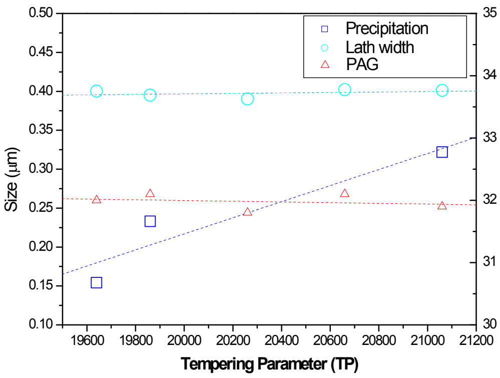

Fig. 1 Tempering parameter with the lath width, prior austenite grain size, and precipitation mean size at 1050℃

Fig. 2 shows the hardness change of HT9 with the heat treatment conditions. The hardness was shown to increase with increase in normalizing temperature. A tensile test was carried out at room temperature and high temperature (650℃) to evaluate the tensile properties of the heat treated specimen under the same conditions. The tensile test was carried out at room temperature and high temperature (650℃). The yield stress shows a tendency to decrease proportionally.

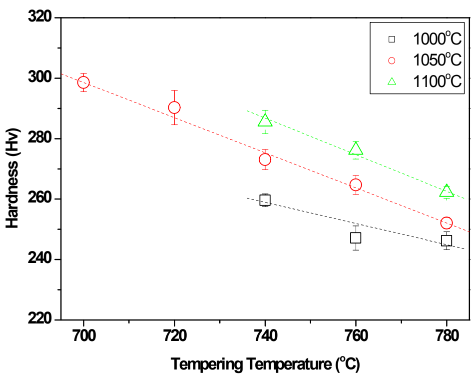

Fig. 2 Hardness change of HT9 with heat treatment condition

Yield stress was shown to increase with an increase in normalizing temperature. These tendencies were also represented at high temperature.

3.2 Aging condition on the microstructure and mechanical property

An HT9 cladding tube was a typical tempered lath Martensite structure, and the PAG size was equal with an increasing normalizing temperature. The lath width increased with an increase in the aging time. Because the cell structures were partially formed by a dislocation recovery inside the lath Martensite structure.Fig.3 shows the carbon exacted replica of HT9 aging condition.

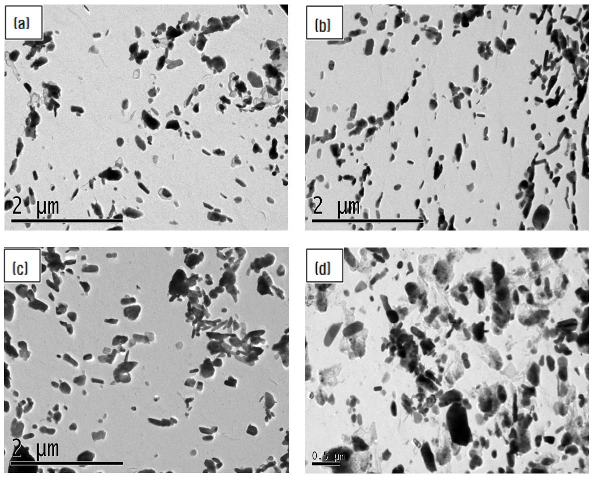

Fig. 3 Carbon exacted replica of HT9 aging conditions: (a) 500 hr, (b) 1000 hr, (c) 3000 hr, and (d) 7000 hr

The precipitate size increased with an increase in aging time. In particular, the size of the precipitates coarsen after 3000hours. Fig. 4 shows the hardness change of HT9 with the aging conditions. The hardness was shown to decrease with an increase in the aging time. A tensile test was carried out at room temperature and a high temperature (650℃) to evaluate the tensile properties of the aged specimen under the same conditions. A tensile test was carried out at room temperature and a high temperature (650℃).

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Because the precipitate growth rate and recovery speed decrease, the width of the mechanical properties are considered to be reduced.

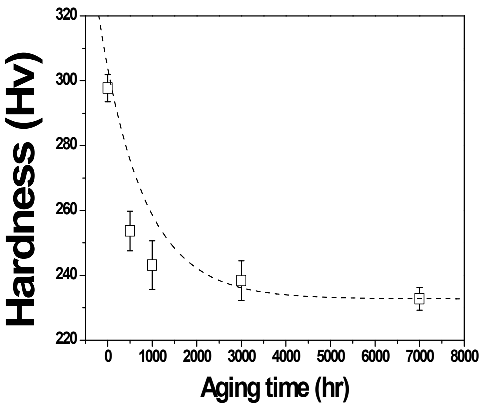

Fig. 4 Hardness change of HT9 with aging time

4. Conclusions

Normalization of HT9 within a temperature range of 1000-1100℃ has contributed to the prior austenite grain size. The yield stress and hardness increase. Tempering of HT9 within the temperature range of 700-780℃ contributed to the increase in precipitate size, and the decrease in yield stress and hardness. An empirical equation for the mechanical properties of HT9 was suggested as a function of the microstructure and Hollomon-Jaffe tempering parameter.

The results show that the size of the carbide and lath increased after aging, whereas the size of the prior austenite grain was not changed. Both the strength and hardness were decreased with aging, and this tendency saturated after 3000 hours of aging. Based on the experimental results, the tempering parameter was modified and discussed along with the aging conditions.

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