Effect of B and N on microstructure of modified 9Cr-2W steel during Aging and Creep

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1. Introduction

Generation IV reactors are found in modern types of nuclear power plants, producing reliable and inexpensive energy in safe and proliferation-resistant reactors. A Generation IV SFR (sodium-cooled fast reactor) uses liquid sodium as a coolant and causes nuclear fission by means of fast neutrons [1]. Because of the high operating temperature in the SFR (up to 650°C), the fuel cladding tube is one of the most important safety barrier in such fission nuclear reactors. Thermal creep and void swelling occurs from fission gas at high temperature during operation. 9~12% Cr ferritic-martensitic (F-M) steels are considered to be ideal candidates for fuel cladding tubes due to their high thermal conductivity, low thermal expansion, good oxidation, corrosion resistance at elevated temperature, and high resistance to void swelling [2]. This study used TEM to examine the growth and generation of precipitates across the change of minor elements (B and N) resulting from aging and creep.

The growth of precipitates by the microstructural degradation was evaluated under a creep and aging environment. The role of B and N on the microstructural change during aging and creep was investigated in this study.

2. Experimental Procedure

2.1. Experimental Material

The chemical composition of the two different kinds of modified 9Cr-2W steel, named alloy B and alloy N, are shown in Table 1. The modified 9Cr-2W steel was melted in a vacuum induction melting (VIM) in mother tubes with 19.05 mm outer diameter, 1.24mm thickness, formed after undergoing a high-temperature extrusion process. And then the modified 9Cr-2W steel tubes underwent five cold drawing processes and subsequent heat treatment.

Alloy B 0.07 8.89 1.93 0.02 0.013 Bal. Alloy N 0.063 9.09 2.01 0.076 0.004 Bal.	•	С	Cr	W	Ν	В	Fe
Alloy N 0.063 9.09 2.01 0.076 0.004 Bal.	-	0.07	8.89	1.93	0.02	0.013	Bal.
	Alloy N	0.063	9.09	2.01	0.076	0.004	Bal.

2.2. Aging Conditions

The mother tubes for aging test were normalized at $1,038^{\circ}$ C for 6 minutes, followed by air cooling to room temperature. Tempering treatment of the normalized specimens was carried out at 760°C for 40 minutes, followed by air cooling to room temperature. Aging specimens of 8 x 25.4 mm and 1.2 mm thickness were taken from a mother tube. Aging test specimens were sealed in quartz tubes in an atmosphere of argon gas, and underwent long-term thermal treatment for a maximum of 7,000 h at 650°C, which represents the peak temperature condition of SFR.

2.3. Creep conditions

The Modified 9Cr-2W steel tubes underwent five cold drawing processes and subsequent heat treatment. Tubes for creep specimens with 7.4 mm outer diameter, 0.5 mm thickness, and 3000 mm length were manufactured. Tubes were normalized at 1,038°C for 6 minutes, followed by air cooling to room temperature. Tempering treatment of the normalized specimens was carried out at 760°C for 40 minutes, followed by air cooling to room temperature out at 760°C for 40 minutes, followed by air cooling to room temperature. Creep tests carried out under constant load conditions and were pressurized to 240MPa, 180MPa, 160MPa, and 140MPa (hoop stress) at 650°C, respectively, and rupture time was measured.

3. Result and Discussion

Alloys B and N were found to have a lot of precipitates, such as $M_{23}C_6$, MX. Alloy N shows that needle-shaped Cr_2N was observed. Precipitate such as $M_{23}C_6$ in alloy B and N was found at along prior austenite grain boundary or martensite lath boundary, while MX and Cr_2N existed at the martensite lath boundary. Alloys B and N, Laves phase was observed, which takes the form of (Fe, Cr)₂W. Fig. 1 shows the variation of Cr/Fe ratio in $M_{23}C_6$ precipitate during aging and creep test. The tempering temperature of alloy B and N was 760°C, which is higher than aging temperature, 650°C. Thus, the Cr/Fe ratio of initially precipitated $M_{23}C_6$ in 9Cr-2W steel after tempering is formed having a stoichiometric value of 2.1 with a certain period of time, whose chemical composition does not change after tempering [3]. In 650°C aging, Cr/Fe ratio in $M_{23}C_6$ begins to change after 1000 h. In the case of creep, the Cr/Fe ratio of $M_{23}C_6$ rapidly increases when compared to the aging test because the creep deformation accelerates the diffusion of carbide in Cr.



Fig. 1 Variation of Cr/Fe ratio in $M_{23}C_6$ during aging and creep at $650^{\circ}C$

The role of B and N on the size of precipitates in the creep and aging specimens were investigated using an image analyzer. Fig. 2 shows precipitate size measured by extraction carbon replica using an image analyzer. The growth of precipitates takes place during aging and



Fig. 2 $M_{23}C_6$ size of alloy B and alloy N during aging and creep at 650°C

creep at high temperature. Initial precipitates show that alloy B bigger in size than alloy N. In the case of aging, the precipitate sizes are kept constant up to 500 h at 650° C, and then they increase after 500 h. From Fig. 2, it was observed that the rate of precipitate growth varies significantly with the alloy type after the aging time of

3000 h. In the case of creep, the precipitate size greatly increases with the small amount of stress.

4. Conclusions

This study analyzed the effects of boron and nitrogen, which add to improve the performance of nuclear fuel cladding tubes for SFR, specifically in relation to thermal stability of precipitates resulting from aging and creep conditions.

- The rate of precipitate growth differs significantly with the alloy type after the aging time of 3000 h. In the case of creep, the precipitate size greatly increases with the small amount of stress.
- 2) The Cr/Fe ratio in the creep test increased faster than in the aging test, which means that the diffusion of Cr was faster in the presence of stress. The addition of B reduces the coarsening rate of Ostwald ripening of $M_{23}C_6$ carbides near prior austenite grain boundaries during creep.
- Alloy B has a constant size of M₂₃C₆ after 1000 h, so rupture time increases due to the pinning effect of small M₂₃C₆. In terms of this, alloy B has a longer rupture time than alloy N.

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