

## Effects of Boron Addition on Creep and Microstructure in 9Cr-0.5Mo-2W-V-Nb-B Steels for a SFR Fuel Cladding

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### 1. Introduction

Sodium-cooled fast reactor(SFR) is one of the prospective nuclear systems for the future generation (Gen-IV) systems. The core materials in the Gen-IV SFR are subjected to a high fast nuclear fluence ( $\geq 200\text{dpa}$ ) and a high irradiation temperature ( $\geq 600^\circ\text{C}$ ) in which atomic displacements take place. Ferritic/martensitic steels are still being considered for a core components application to these fuel cladding and ducts due to their superior thermal and irradiation stabilities, even for the harsh operational envisioned in the Gen-IV SFR designs [1]. So, the development of advanced ferritic/martensitic steels with an excellent creep resistance at higher temperatures over  $600^\circ\text{C}$  is needed for the fuel cladding and ducts for the Gen-IV SFR. In order to overcome the drawbacks of ferritic/martensitic steels under a harsh condition, a new project was launched to develop an advanced core material for the Gen-IV SFR in Korea [2].

Creep strength of the ferritic/martensitic steels has been improved by their martensitic lath structure, the precipitation strengthening effects of  $\text{M}_{23}\text{C}_6$  carbides and MX carbonitrides ( $\text{M}=\text{Nb}, \text{V}, \text{Cr}$ , and  $\text{X}=\text{C}, \text{N}$ ) and the solid solution strengthening effects of Mo and W in the matrix [3]. Especially, the precipitation strengthening effect of MX is important because its coarsening rate is small and a fine particle size is maintained for a long-term creep exposure [4]. Many researchers have pointed out that the features and the role of MX can affect the creep behaviors of these steels [5]. Z-phase formation from MX-type precipitates has been proposed as a degradation mechanism for a long-term creep regime [6]. Some studies have been performed to verify the boron addition effects on the creep properties of ferritic/martensitic steels by the formation of boron-incorporated  $\text{M}_{23}\text{C}_6$  carbides [7].

In this study, it was designed so that 0.2 wt.% V and 0.2 wt.% Nb were involved in a 9 wt.% chromium ferritic/martensitic steel so as to increase the number of MX precipitates and the boron was intentionally added to the steel to improve its creep resistance. The creep and microstructures of the 9Cr-0.5Mo-2.0W-V-Nb-B steels were characterized and analyzed on the basis of the experimental results.

### 2. Experimental Procedure

The chemical compositions of the two experimental steels used in this study are shown in Table 1. Carbon and nitrogen contents were almost 0.1 wt.% and 0.08 wt.%, respectively. And the boron concentration of the B003 steel was 170 ppm. 30kg ingots were melted by a vacuum induction melting(VIM) method and hot-rolled to a 15mm thickness at  $1150^\circ\text{C}$ . The hot-rolled specimens were austenitized at  $1050^\circ\text{C}$  for 1 hour and then they were tempered at  $750^\circ\text{C}$  for 2 hours. All the specimens were cooled in air at room temperature after the austenitization and tempering treatments. Creep tests were performed at  $650^\circ\text{C}$  under the constant load conditions of 110, 120, 130 and 140MPa.

Table 1 Chemical composition of the experimental steels

Exp. Steels	Composition, wt.%						
	Cr	Mo	W	V+Nb	C	N	B
B001	8.95	0.528	2.13	0.413	0.11	0.08	-
B003	8.95	0.536	1.98	0.403	0.09	0.09	0.017

For the precipitate analysis of the as-austenitized/tempered and crept samples, precipitates were detected by using a transmission electron microscope, a scanning transmission electron microscope and a secondary ion mass spectrometry, which was attached to an energy dispersive X-Ray spectroscopy. A comparison method between Cr and V maps was utilized to distinguish the Z-phases from the MX carbonitrides. Extracted carbon replicas were prepared by the evaporation of carbon on a polished and etched sample surface followed by a dissolution of the metallic matrix in an etchant of 10% HCl-90% methanol at a voltage of 20V at  $25^\circ\text{C}$ . Optical microscope and TEM were also utilized to observe the matrix microstructures, and microhardness tests were performed with a load of 500g to evaluate the hardness decrease after a creep exposure.

### 3. Results and Discussion

#### 3.1 Creep Properties

Fig.1 presents the creep rupture stress with the time to a rupture after performing the creep tests at  $650^\circ\text{C}$  for the two experimental steels, together with a commercial ASTM Gr.92. Compared with the references of Gr.92, the creep rupture stresses of the experimental steels of B001 and B003 were higher than those of the references. The improvement of the creep rupture stress

could be attributed to the higher nitrogen content in the experimental steel. The increase of both the Nb and N contents in the B001 steel could determine the amount of V-rich MX as well as Nb-rich MX precipitates.

The creep rupture stress of the B003 steel was higher than that of the B001 steel. The chemical composition of the two experimental steels was almost the same except for the boron content. Boron of 170 ppm was only added to the B003 steel. The presence of boron in the carbides could retard the coarsening rate of these carbides and the recovery of a dislocation substructure during a long-term creep exposure. The beneficial effect of a boron addition to the B003 steel was observed in the creep environments at 650°C.

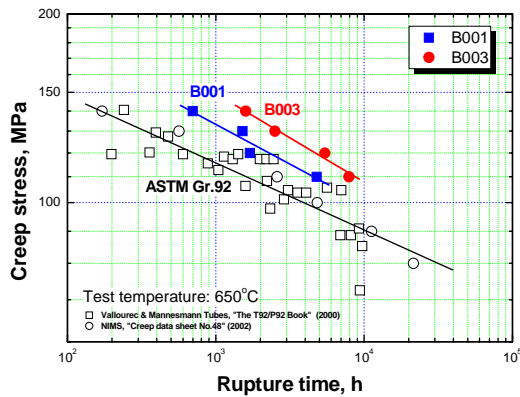


Fig. 1 Creep stress of the experimental steels with the rupture time

### 3.2 Microstructural Properties

Two experimental steels show a typical tempered martensitic microstructure after the normalizing and tempering treatments. The martensite lath width showed a tendency to decrease in the boron-added B003 steel when compared with the B001 steel. The  $M_{23}C_6$  carbides were formed as major precipitates around the prior-austenite and lath boundaries. Small content of MX-type carbonitride precipitates (Nb-rich MX and V-rich MX) was observed on both boundaries and in their interiors. Additionally,  $M_2X$  precipitates were also detected on both boundaries in the two experimental steels.

The elemental distribution in the B003 steel before the creep tests was observed by using STEM and SIMS. From the STEM images, elements of Cr, Fe, Mo, W, V, C, and N, except for Nb, were concentrated along the prior-austenite boundaries. It was thought that the  $M_{23}C_6$ , V-rich MX, and  $M_2X$  precipitates were located along the prior-austenite boundaries. But the Nb-rich MX precipitates were observed in the interior of the prior-austenite boundaries. This could have resulted from the fact that the Nb-rich MX precipitates were mainly formed by the insoluble Nb during the normalization at less than 1250°C. But the  $M_{23}C_6$ , V-rich MX and  $M_2X$  phases were nucleated and grown during the tempering as well as the normalizing

treatment. The boron in the B003 steel would be distributed at the interface between the  $M_{23}C_6$  carbides and the matrix, and within the carbides. The boron mapping by SIMS also indicates that the boron is distributed along the prior austenite boundaries. The boron addition could suppress the coarsening of the  $M_{23}C_6$  carbides and the nucleation of the Laves phases that precipitate near the prior-austenite boundaries during a long-term creep exposure. This would improve the creep resistance of the boron-added B003 steel when compared with that of the B001 steel.

## 4. Conclusions

It was summarized that the present steels of B001 and B003 had a higher creep resistance than the commercial ASTM Gr.92 steel. It was thought that the higher niobium and nitrogen contents in the B001 and B003 steels could enhance the creep rupture stress at 650°C since the number density of the Nb-rich MX precipitates would increase. And the boron addition to the B003 steel could improve its creep properties due to a reduction of the martensitic lath width as well as an  $M_{23}C_6$  carbide stabilization by the boron addition around the prior austenite grain boundaries.

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