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Observation of TEM microcstructure for β-quenched Zr-Nb alloys

Yeon-Soo Na*, Tae-Sik Jung, Yong-Kyoon Mok, Jae-Ik Kim
KEPCO Nuclear Fuel, 1047 Daedukdaero, Yuseong-gu, Daejeon, Korea, 305-353
**Corresponding author: ysna@knfc.co.kr*

1. Introduction

Zirconium based alloy has been successfully used as fuel cladding in nuclear reactor environements for many years [1]. However, as PWRs tend to be operated in more severe nuclear reactor environments, the higer corrosion resistant Zr alloys have been continuously developed [2]. At KEPCO NF(KEPCO Nuclear Fuel), the advanced Zr-Nb alloys which were systematically controlled by alloying elements and heat treatment are developing with the aim of more superior Zr-Nb alloys with various properties such as corrosion, creep and fatigue resistance than commercial zirconium alloys.

The aim of this study is to investigate the precipitates and microstructures of Zr-Nb alloys containing X-element, and the characteristics of the precipitates were analyzed by TEM (Transmission Electron Microscope).

2. Experimental procedure

The chemical compositions of the Zr alloys used in this study are shown Table 1. The experimental Zr alloys can be classified into two groups according to the added X-elements; A: Zr-Nb-Al-Cu-X and B: Zr-Nb-Cu-X alloys. The experimental alloys were prepared by VAR (Vacuum Arc Remelting). As shown in Fig. 1, ingots were remelted by at least three times to improve chemical homogeneity. The manufactured arc-remelted button ingots was about 68mm in diameter and 10mm in thickness, and weights about 300g. The button ingots were β-heat treatment to achieve homogenized composition at 1020℃ for 30min in a furnace.

The microstructures of the β-quenched Zr-Nb alloys were characterized by TEM to ensure that the alloys had precipitates containing X-element, and the compositions of precipitates were measured by using energy-dispersive spectroscopy (EDS). Specimens for TEM observation were prepared by a twin-jet polishing with a solution of 10 vol.% $HClO_3$ and 90 vol.% C_2H_5OH after mechanical polishing to 50 ㎛ in thickness by SiC paper.

Table 1. Chemical composition of Zr-Nb alloys containing X-element in this study (wt.%)

ID	Nb	Al	Cu	X	Zr
A	1.1	0.05	0.05	0.05	bal.
B	1.1	-	0.05	0.05	bal.

Fig. 1 Button ingot manufactured by VAR

3. Results and Discussion

3.1 Observation of TEM microstructure for Zr-Nb-Al-Cu-X alloy

Fig. 2 shows the TEM microstructures, diffraction pattern and EDS data of Zr-Nb-Al-Cu-X alloy. From this figure, widmanstatten structure with lamella morphology in Zr matrix regardless of water quenching condition was observed [3]. Also, from the results of diffraction pattern in TEM-EDS, white band shaped α-Zr phase and black line shaped β-Zr phase were ranged repeatedly.

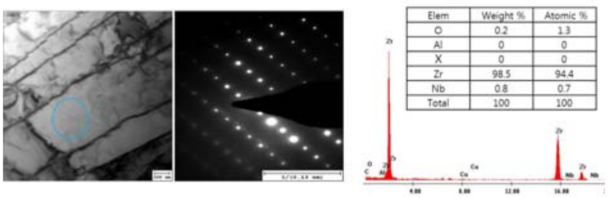

Fig. 2 TEM microstructure, diffraction pattern and EDS data of β-quenched Zr-Nb-Al-Cu-X alloys

The precipitates of Zr-Nb-Al-Cu-X alloy were also observed by using TEM. From the Fig. 3, β-X precipitates of BCC (Body-centrered cubic) structure in Zr matrix was inhomogeneously formed. This particle size was approximately 2~3㎛, and the biggest particle size was 5㎛. However, the precipitates as β-Nb and Zr(Nb,X) type were not observed in Zr matrix. It was considered that the Nb element was supersaturated in α-Zr matrix without the formation of second phase or precipitates because the cooling rate was so fast by water quenching [4].

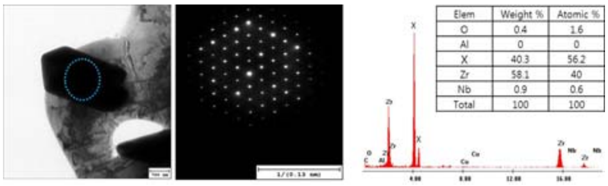

Fig. 3 BCC β-X precipitates of β-quenched Zr-Nb-Al-Cu-X alloy

3.2 Observation of TEM microstructure for Zr-Nb-Cu-X alloy

Fig. 4 and 5 show the TEM microstructures, diffraction pattern and EDS data of Zr-Nb-Cu-X alloy, respectively. From this figure, as the Zr-Nb-Al-Cu-X alloys, the widmanstatten structure and BCC β-X precipitates were observed in Zr matrix and it was inhomogeneously distributed in Zr matrix. This particle size approximately was 2~3 ㎛, and the biggest particle size was 4 ㎛. Therefore, from the above results, it is considered that Zr-Nb alloy containing X-element for working process as a hot rolling and cold rolling will be able to form the precipitates as β-X and Zr(X,Nb) types.

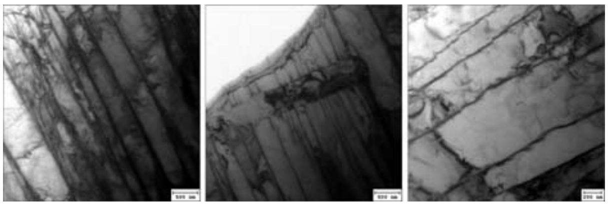

Fig. 4 TEM microstructure of β-quenched Zr-Nb-Cu-X alloy

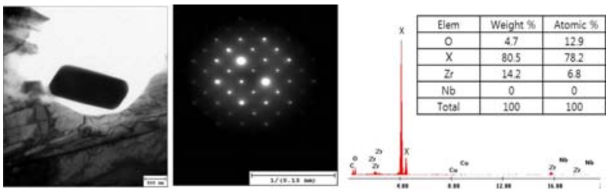

Fig. 5 BCC β-X precipitates of β-quenched Zr-Nb-Cu-X alloy

4. Conclusions

In order to investigate the precipitates and microstructures of Zr-Nb alloys containing X-element, TEM microstructure observation and the precipitates analysis were performed by using a TEM equipped with EDS. The precipitates containing X-element in both Zr-Nb alloys were observed. Therefore, it is considered that Zr-Nb alloy containing X-element for working process as a hot rolling and cold rolling will be able to form the preciptation containing X-element as β-X and Zr(X,Nb) types.

Acknowledgements

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