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# Aging Effect on Corrosion and Microstructure of Zr-1.5Nb-0.4Sn-0.2Fe-0.1Cr Alloy

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## 1. Introduction

A variety of Nb-containing Zr alloys have been developed as the fuel cladding materials for several decades, since the corrosion resistance was improved by the Nb addition as an alloying element [1-3]. However, the corrosion behavior of Nb-containing Zr alloys, with especially high Nb content more than 1.0 wt %, is greatly affected by thermal processing [4, 5]. They reported that the corrosion properties of the Nb-containing Zr alloys were considerably affected by the microstructure conditions such as the Nb-concentration in the matrix and the second phase types. On the other hand, the aging effect is generally introduced to improve the corrosion resistance for Zr-based alloys, and this improved corrosion resistance was shown to be related to the microstructure changes.

The Zr-1.5Nb-0.4Sn-0.2Fe-0.1Cr composition for applying the fuel cladding material was newly developed at KAERI after a systematic control of the alloying elements. Since this new alloy system contains an especially high Nb content, it is necessary to study the aging effect on the corrosion and microstructure change to increase the corrosion resistance. Therefore, the purpose of this work is to elucidate the optimum aging conditions for the Zr-1.5Nb-0.4Sn-0.2Fe-0.1Cr alloy.

## 2. Methods and Results

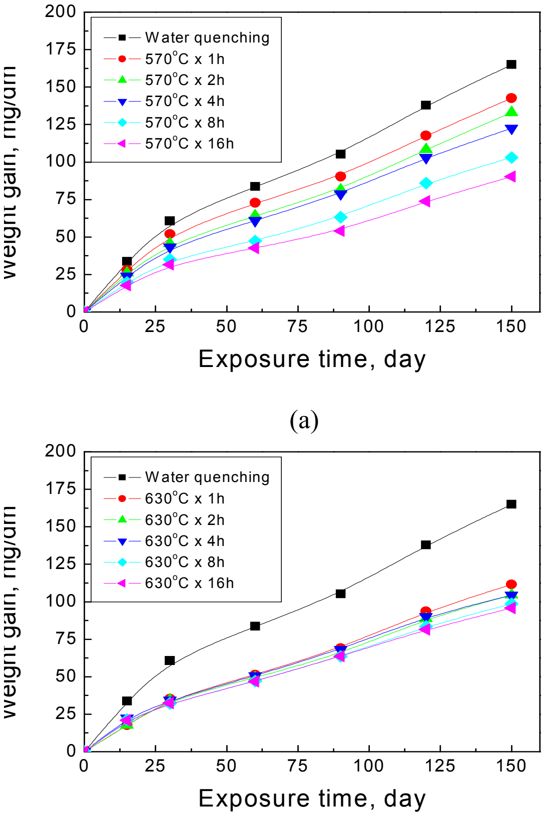
The manufacture of the Zr-1.5Nb-0.4Sn-0.2Fe-0.1Cr alloy involved a vacuum sequence in which the alloy was melted four times to promote the homogeneity of the alloying element. The melted ingot was quenched in a water from the β solution region at 1050℃. The quenched ingot was cut in to dimensions of 16 mm x 28 mm x 12 mm and then aged in a vacuum environment at various temperatures and times as shown in Table 1.

Table 1 Isothermal aging conditions of Zr-1.5Nb-0.4Sn-0.2Fe-0.1Cr alloy.

| Aging temperature ($^o$C) | Aging time (h) |
|---|---|
| 660 | 1, 2, 4, 8, 16 |
| 630 | 1, 2, 4, 8, 16 |
| 600 | 1, 2, 4, 8, 16 |
| 570 | 1, 2, 4, 8, 16 |
| 540 | 1, 2, 4, 8, 16 |

The corrosion test was performed in a static autoclave with a 360℃ water environment under saturated pressure of 18.9 MPa. The size of corrosion test specimens was of 15 mm x 25 mm x 1.0 mm after mechanically ground with 1200 grit SiC paper for the aged samples. The ground specimens were then pickled in a mixed solution of $H_2O$ (40 vol. %), $HNO_3$ (30 vol. %), HCl (25 vol. %) and HF (5 vol. %). The corrosion resistance was evaluated by measuring the weight gain of the corroded samples after suspending the corrosion test at a periodic interval.

The microstructure observation for the each aged sample was performed by using transmission electron microscope (TEM) equipped with energy dispersive spectra (EDS) capabilities. The specimens for the TEM observation were prepared by being mechanically thinned to about 70 μm and then being subjected to a twin-jet polishing method with a mixed solution of $C_2H_5OH$ (90 vol. %) and $HClO_4$ (10 vol. %).



(a)

(b)

Fig. 1 Corrosion behavior of Zr-1.5Nb-0.4Sn-0.2Fe-0.1Cr alloy aged at 570℃ (a) and 630℃ (b) after β-quenching

Fig. 1 shows the corrosion weight gain of Zr-1.5Nb-0.4Sn-0.2Fe-0.1Cr alloy as a function of exposure time. The weight gain of the aged Zr-1.5Nb-0.4Sn-0.2Fe-

0.1Cr alloy was increased with increasing the exposure time. The weight gain of all aged specimens in both 570℃ and 630℃ was lower than that of water quenched specimens. So, it is known that the corrosion resistance of the Zr-1.5Nb-0.4Sn-0.2Fe-0.1Cr alloy is increased by applying the aging process after the water quenching. After the aging at 570℃, the corrosion weight gain was gradually decreased with increasing the aging time. Whereas, the corrosion weight gain was considerably decreased in all the aged specimens after the aging at 600℃, and the weight gain was not changed with the difference aging times in this temperature.

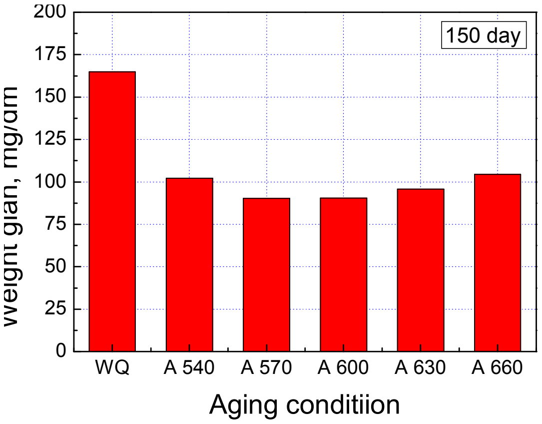


Fig. 2 Corrosion weight gain of Zr-1.5Nb-0.4Sn-0.2Fe-0.1Cr alloy with the aging temperatures

Fig. 2 shows the comparison of corrosion weight gain after 150 day test. The value of the weight gain for the aged specimens was selected from the 16 h aging condition at each aging temperature, since the aging time of 16 h showed the lowest weight gain. The corrosion resistance of all aged specimens for 16 h was increased up to about 60% when compared to the water quenched specimens. Since, the lowest weight gain was shown when the specimens were annealed at 570 ℃ and 600 ℃, the optimum temperature for the aging of Zr-1.5Nb-0.4Sn-0.2Fe-0.1Cr alloy was ranged from 570 to 600 ℃.

The TEM microstructure analysis for the aged specimens is shown in Fig. 3. The second phases were precipitated in the matrix in both the 570 and 640℃ aging. But the size and type of the second phase were shown differently by the aging temperatures. The size of the second phases in the matrix was smaller in the 570℃ aged specimen than in the 640℃ aged specimen. From the analysis of the second phase, $Zr(NbFeCr)_2$ phase was formed after the 570℃ aged specimen, whereas, two types of β-Zr and $Zr(NbFeCr)_2$ phases were formed after the 570℃ aged specimen. So, it is known that the second phase characteristics could be changed by the aging temperature. However, the second phase characteristics have not considerably effected on the corrosion resistance, since the corrosion weight gain of the aged specimens between 570℃ and 630℃ was similar. From this, it could be assumed that the major effect on the improved corrosion resistance with the aging of Zr-1.5Nb-0.4Sn-0.2Fe-0.1Cr alloy was related to the decrease of the supersaturated element concentrations of Nb, Fe, and Cr in the α matrix.

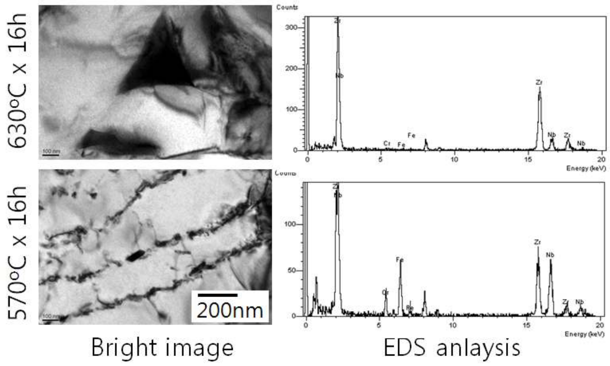


Fig. 3 TEM analysis of the Zr-1.5Nb-0.4Sn-0.2Fe-0.1Cr alloy aged at 570℃ and 630℃ for 16h

## 3. Conclusions

The corrosion resistance of the Zr-1.5Nb-0.4Sn-0.2Fe-0.1Cr alloy was increased by applying the aging process after the water quenching. And the optimum temperature for the aging of Zr-1.5Nb-0.4Sn-0.2Fe-0.1Cr alloy was ranged from 570 to 600 ℃.

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